

Interlaboratory Proficiency Test 07/2019

Gross and net calorific values in fuels

**Mirja Leivuori, Eliisa Hatanpää, Riitta Koivikko,
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ABSTRACT

Interlaboratory Proficiency Test 07/2019

Profest SYKE arranged the proficiency test (PT) for the measurements of the gross and net calorific value, the content of ash, carbon, nitrogen, hydrogen, moisture, sulphur, and volatile matter in peat, wood pellet (not sulphur) and coal samples in August-September 2019. In total, there were 34 participants in the PT. The participants could also estimate the emission factor for the peat and coal samples.

The robust mean or the median of the results reported by the participants was used as the assigned value for measurements. The performance evaluation was based on the z scores. In total, 90 % of the reported results were satisfactory, when the deviation of 1–30 % from the assigned value was accepted. For the gross calorific value measurements 93 % of the peat sample results, 76 % of the wood pellet sample results, and 86 % of the coal sample results were satisfactory. For the net calorific value measurements 82 % of the peat sample results, 88 % of the wood pellet results, and 93 % of the coal sample results were satisfactory. The performance evaluation was not done for the measurements of M_{ad} in all samples, N_d in the wood pellet sample, and emission factor in peat and coal sample.

Warm thanks to all the participants in this proficiency test!

Keywords: Proficiency test, interlaboratory comparison, coal, peat, wood pellet, calorific value, emission factor, ash, moisture, carbon, sulphur, nitrogen, hydrogen, volatile matter, environmental laboratories

TIIVISTELMÄ

Laboratorioiden välinen pätevyyskoe 07/2019

Profest SYKE järjesti elo-syyskuussa 2019 pätevyyskokeen kalorimetrisen ja tehollisen lämpöarvon sekä tuhkan, vedyn, hiilen, typen, rikin, haihtuvien yhdisteiden ja kosteuden määrittämiseksi turpeesta, puupelletistä (ei rikkiä) ja kivihiilestä. Lisäksi osallistujilla oli mahdollisuus arvioida ja/tai laskea turve- ja kivihiilinäytteiden päästökerroin. Pätevyyskokeessa oli yhteensä 34 osallistujaa.

Testisuureiden vertailuarvoina käytettiin osallistujatulosten robustia keskiarvoa tai mediaania. Pätevyyden arviointi tehtiin z-arvojen avulla. Koko tulosaineistossa hyväksyttäviä tuloksia oli 90 %, kun vertailuarvosta sallittiin 1–30 % poikkeama. Kalorimetrisen lämpöarvon tuloksista oli hyväksyttäviä 93 % (turve), 76 % (puupelletti) ja 86 % (kivihiili). Tehollisen lämpöarvon tuloksille vastaavat hyväksyttävien tulosten osuudet olivat 82 % (turve), 88 % (puupelletti) ja 93 % (kivihiili). Tulosten arviointia ei tehty testinäytteiden kosteuspitoisuuden määrittämiselle, puupelletin typen määrittämiselle ja päästökertoimelle turve- ja hiilinäytteissä.

Kiitos pätevyyskokeen osallistujille!

Avainsanat: pätevyyskoe, vertailumittaus, kalorimetrisen lämpöarvo, tehollinen lämpöarvo, päästökerroin, tuhka, kosteus, hiili, rikki, typi, haihtuvat yhdisteet ja vety, turve, puupelletti, hiili, ympäristölaboratoriot

SAMMANDRAG

Provningsjämförelse 07/2019

Profest SYKE genomförde i augusti-september 2019 en provningsjämförelse som omfattade bestämningen av kalorimetriskt och effektivt värmevärde, svavel, väte, kol, kväve, askhalt, flykthalt och fukthalt i torv, träd pellet (inte svavel) och stenkol. Det var en möjlighet att beräkna emissionfaktor i torv och stenkol prover. Totalt 34 deltagarna deltog i jämförelsen.

Som referensvärde för analyternas koncentration användes det robusta medelvärde eller den medianen av deltagarnas resultat. Resultaten värderades med hjälp av z värden. I jämförelsen var 90 % av alla resultaten acceptabel värderades, när en total deviation på 1–30 % från referensvärdet tilläts. Av det kalorimetriska värmevärdet var 93 % acceptabla (torv), 76 % (trädpellet) och 86 % (stenkol). För resultaten av det effektiva värmevärdet var 82 % (torv), 88 % (trädpellet) och 93 % (stenkol) acceptabla. Det var inte gjorts värdering till fukthalt i alla prover, beräkning av väte i torv provet, nitrogen i trädpellet och emissionfaktor i torv och stenkol provet.

Ett varmt tack till alla deltagarna i testet!

Nyckelord: provningsjämförelse, kalorimetriskt och effektivt värmevärde, emissionfaktor, svavel, väte, kol, nitrogen, askhalt, flykthalt fukthaltstenkol, torv, träd pellet, miljölaboratorier

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1 Introduction

Proftest SYKE carried out the proficiency test (PT) for analysis of gross and net calorific value in fuels in August-September 2019 (CAL 07/2019). In the PT, gross and net calorific value, C_d , S_d , H_d , N_d , moisture content of the analysis sample ($M_{ad,d}$), ash content as well as volatile matter (V_{db}) were tested in peat, wood pellet (not S) and coal samples. Additionally, the participants were asked to estimate the emission factors (EF) for the peat and coal samples.

Finnish Environment Institute (SYKE) is appointed National Reference Laboratory in the environmental sector in Finland. The duties of the reference laboratory include providing interlaboratory proficiency tests and other comparisons for analytical laboratories and other producers of environmental information. This proficiency test has been carried out under the scope of the SYKE reference laboratory and it provides an external quality evaluation between laboratory results, and mutual comparability of analytical reliability. The proficiency test was carried out in accordance with the international standard ISO/IEC 17043 [1] and applying ISO 13528 [2] and IUPAC Technical report [3]. The Proftest SYKE is accredited by the Finnish Accreditation Service as a proficiency testing provider (PT01, ISO/IEC 17043, www.finas.fi/sites/en). The organizing of this proficiency test is included in the accreditation scope of the Proftest SYKE.

2 Organizing the proficiency test

2.1 Responsibilities

Organizer

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The responsibilities in organizing the proficiency test

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Riitta Koivikko	substitute for coordinator
Keijo Tervonen	technical assistance
Markku Ilmakunnas	technical assistance
Sari Lanteri	technical assistance

Analytical expert

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Subcontracting

Homogeneity testing: FTF Fuel Testing Finland Oy (T329, www.finas.fi/sites/en, formerly Eurofins Environment Testing Finland Oy, Vantaa)

The wood pellet samples were homogenated and divided into sub-samples at the laboratory of KVVY Tutkimus Oy (Tampere, Finland, T064, www.finas.fi/sites/en).

2.2 Participants

In total 34 laboratories participated in this proficiency test, of which 11 were from Finland and 23 from abroad (Appendix 1).

Altogether 76 % of the participants used accredited analytical methods at least for a part of the measurements. The samples were tested at the laboratory of FTF Fuel Testing Finland Oy and their participant code is 13 in the result tables.

2.3 Samples and delivery

Three different fuel samples were delivered to the participants: peat (B1), wood pellet (B2) and coal (K1) samples. Gross ($q_{v,gr,d}$) and net ($q_{p,net,d}$) calorific value, C_d , S_d , H_d , N_d , moisture content of the analysis sample ($M_{ad,d}$), ash content as well as volatile matter (V_{db}) were tested in peat, wood pellet (not S) and coal samples.

The wood pellet sample (B2) was provided by Vapo Oy and it was pre-treated (grinding) by Eurofins Labtium Ltd, Jyväskylä (T025, www.finas.fi/sites/en). Wood pellet samples were homogenated and divided into sub-samples at KVVY Tutkimus Oy in Tampere (T064, www.finas.fi/sites/en). In this PT the peat sample B1 from the PT CAL 05/2009 [4] and coal sample K1 from the PT CAL 05/2010 [5] were used. The samples B1 and K1 were re-homogenated and divided into subsamples in the laboratory of Proftest SYKE.

The laboratory of FTF Fuel Testing Finland Oy (T329, www.finas.fi/sites/en) tested all samples. The sample preparation is described in details in the Appendix 2.

In the cover letter delivered with the samples, the participants were instructed first to store the samples closed for one day after their arrival and then to measure the moisture content of the analysis sample (M_{ad}) as the first measurement. The samples were instructed to be homogenized before the measurements and to be stored in a dry place at room temperature. Further, the sample moisture content was instructed to be analyzed on every measurement day. This was important as it eliminates the influence of humidity on the measurements.

Participants could also estimate/calculate the emission factor (as received), EF, for peat and coal samples. For this estimation/calculation the total moisture contents of the samples as received (M_{ar}) were given:

- peat B1 50.8 %,
- coal K1 11.2 %

The samples were delivered to the participants on 23 August 2019 and they arrived to the participants mainly latest on 30 August 2019. One participant informed the arrival of the samples on 13 September 2019, but the tracking system of the delivery courier showed the sample arrival to the pick-up location on 30 August 2019.

The samples were requested to be measured and the results to be reported latest on 20 September 2019. The results were mainly reported within the requested time, one participant reported the results on 23 September 2019. The preliminary results were delivered to the participants via Proftest [WEB](#) and email on 26 September 2019.

2.4 Homogeneity studies

Homogeneity of the sample B2 was tested by measuring the gross and net calorific value and ash content as duplicate determinations from five subsamples and from three subsamples for the samples B1 and K1 (Appendix 3). Moreover, the other measurands were tested from two subsamples as duplicate measurements. According to the homogeneity test results, all samples were considered homogenous. Based on the knowledge of the provider the samples have been considered stable during the PT. The peat and coal samples (B1 and K1) were used also in earlier PTs and they were considered to be fit for purpose based on the data from both earlier and current homogeneity test.

2.5 Feedback from the proficiency test

The feedback from the proficiency test is shown in Appendix 4. The comments from the participants mainly dealt with sample delivery and participants' reporting errors. The comments from the provider are mainly focused to the lacking convergence to the given information with the samples. All the feedback is valuable and is exploited when improving the activities.

2.6 Processing the data

2.6.1 Pretesting the data

To test the normality of the data the Kolmogorov-Smirnov test was applied. The outliers were rejected according to the Grubbs or Hampel test before calculating the mean. Also, before the statistical results handling some outliers were rejected in cases where the result differed from the data more than $s_{rob} \times 5$ or 50 % from the robust mean. The rejection of results was partly based on the rather strict requirements for the reproducibility given in the standards for analysis described in the cover letter of the samples. The duplicate results were tested using the Cochran test. If the result was reported as lower than the limit of determination (LOD) or the requested replicate results were not reported, the participant result has not been included in the calculations.

More information about the statistical handling of the data is available in the Guide for participant [6].

2.6.2 Assigned values

Mainly the robust mean was used as the assigned value for measurands of the test samples, when there were at least 12 results ($n_{\text{stat}} \geq 12$). When the robust mean is calculated, the outliers are normally not rejected, but they are iterated before the final calculation of the robust mean. However, in this proficiency test some extreme results were considered as clear outliers and thus rejected. In cases, where the number of results was lower than 12, the median of the reported participants' results was used as the assigned value. For the peat sample B1 the median was used as the assigned value for measurands: C_d , EF, H_d , N_d , $q_{p,\text{net},d}$, S_d , and V_{db} , for the wood pellet sample B2 for the measurands: C_d , H_d , N_d , and V_{db} , and for the coal sample K1 for measurands: EF and N_d . For nitrogen (N_d) in the pellet sample (B2) the informative assigned value is given, but due to the high deviation of results the performance evaluation was not done.

When the robust mean or the median was used as the assigned value, the uncertainty was calculated using the robust standard deviation or the standard deviation [2, 6].

When using the robust mean or the median of the participant results as the assigned value, the expanded uncertainties of the assigned values for calorific values were between 0.2 % and 0.6 %. For the other evaluated measurands the expanded uncertainty varied from 0.2 % to 7.5 % (Appendix 5).

After reporting the preliminary results noticed that one participant was used external laboratory for H_d and N_d results for all tested samples. In the proficiency test is not allowed to use subcontracting laboratory, and thus those results were subtracted from the final database. The assigned value for H_d in the sample B2 was changed from 5.94 to 5.93 w% and for N_d in the sample B1 from 2.70 to 2.71 w%. For other samples there were no changes in the assigned value. **These changes caused no change to the other participants' performance evaluation, but numerical z values have slightly changed.**

2.6.3 Standard deviation for proficiency assessment and z score

The results of this proficiency test were evaluated with the z score. The requirements for the reproducibility of the used standard methods were reported in the cover letter of the samples and they were used to estimate the standard deviation for the proficiency assessment in this PT. Best performance regarding the reproducibility required for the standard methods was for gross calorific values. The standard deviation for the proficiency assessment ($2 \times s_{pt}$ at the 95 % confidence level) was set to 1–30 % depending on the measurements. The standard deviation for the proficiency assessment was not given for analysis moisture content $M_{ad,d}$ (all samples), nitrogen N_d (B2) and emission factor EF (B1, K1), and thus the performance evaluation for the results is not given.

After reporting the preliminary results no changes have been done for the standard deviations of the proficiency assessment values.

When using the robust mean or the median as the assigned value, the reliability was tested according to the criterion $u_{pt} / s_{pt} \leq 0.3$, where u_{pt} is the standard uncertainty of the assigned value and s_{pt} is the standard deviation for proficiency assessment [3]. When testing the reliability of the assigned value the criterion was mainly fulfilled and the assigned values were considered reliable.

The reliability of the standard deviation and the corresponding z score was estimated by comparing the deviation for proficiency assessment (s_{pt}) with the robust standard deviation (s_{rob}) or standard deviation (s , $n_{stat} < 12$) of the reported results (the criteria) [3]. The criterion $s_{rob} \text{ (Or } s) / s_{pt} < 1.2$ was mainly fulfilled.

3 Results and conclusions

3.1 Results

The summary of the results of this proficiency test is presented in Table 1. Explanations to terms used in the result tables are presented in Appendix 6. The results and the performance of each participant are presented in Appendix 7. The reported results with their expanded uncertainties ($k=2$) are presented in Appendix 8. The summaries of the z scores are shown in Appendix 9 and the z scores in the ascending order in Appendix 10. If the participant did not report the requested replicate results for measurands, the evaluation scores are not available. When needed the participant can calculate their own z scores [6].

The robust standard deviations or the standard deviations of the results varied from 0.2 to 12.7 % (Table 1). The robust standard deviation or the standard deviation was lower than 2 % for 50 % of the results and lower than 6 % for 88 % of the results (Table 1). For Ash_d (B2), N_d (B2) and S_d (B1) the robust standard deviation of the results was higher than 6 % (Table 1). The robust standard deviations and the standard deviations were approximately within the same range as in the previous similar proficiency test Profest SYKE CAL 07/2018, where the deviations varied from 0.3 % to 13.3 % [7].

Table 1. The summary of the results in the proficiency test CAL 07/2019.

Measurand	Sample	Unit	Assigned value	Mean	Rob. mean	Median	s_{rob} / s	$s_{rob} \% / s \%$	$2 \times s_{pt} \%$	n_{all}	Acc z %
Ash _d	B1	w%	5.38	5.40	5.38	5.45	0.16	3.0	7	14	92
	B2	w%	0.27	0.28	0.27	0.29	0.03	12.7	30	20	94
	K1	w%	10.8	10.8	10.8	10.8	0.1	0.6	2.5	22	90
C _d	B1	w%	54.7	54.5	-	54.7	0.5	0.8	2.5	6	100
	B2	w%	50.2	50.3	50.0	50.2	0.7	1.5	2.5	10	90
	K1	w%	72.3	72.3	72.3	72.2	0.7	1.0	2.5	15	100
EF	B1	t CO ₂ /TJ	106	106	-	106	1.0	0.7	-	4	-
	K1	t CO ₂ /TJ	94.2	94.2	-	94.2	0.2	0.2	-	7	-
H _d	B1	w%	5.74	5.70	-	5.74	0.21	3.7	9	7	100
	B2	w%	5.93	5.88	5.89	5.93	0.25	4.3	10	10	100
	K1	w%	4.42	4.44	4.42	4.39	0.12	2.8	7	13	92
M _{ad,d}	B1	w%	9.10	9.10	9.10	9.11	0.38	4.2	-	15	-
	B2	w%	8.87	8.88	8.87	8.87	0.24	2.7	-	22	-
	K1	w%	3.47	3.47	3.47	3.45	0.18	5.3	-	24	-
N _d	B1	w%	2.71	2.73	-	2.71	0.08	2.9	10	7	100
	B2	w%	0.08	0.09	0.09	0.08	0.04	38.7	-	9	-
	K1	w%	2.18	2.15	2.16	2.18	0.11	5.3	10	10	89
q _{p,net,d}	B1	J/g	21254	21264	21267	21254	149	0.7	1.5	11	82
	B2	J/g	18821	18820	18821	18821	162	0.9	1.8	17	88
	K1	J/g	28203	28216	28203	28212	151	0.5	1.3	15	93
q _{V,gr,d}	B1	J/g	22471	22466	22471	22484	106	0.5	1.4	14	93
	B2	J/g	20142	20136	20142	20167	143	0.7	1.4	21	76
	K1	J/g	29137	29137	29137	29139	84	0.3	1.0	22	86
S _d	B1	w%	0.20	0.20	0.20	0.20	0.02	8.2	15	10	90
	K1	w%	0.31	0.31	0.31	0.31	0.02	5.2	15	19	89
V _{db}	B1	w%	70.2	70.2	70.1	70.2	0.7	0.9	3	8	88
	B2	w%	85.2	85.1	85.2	85.2	1.2	1.4	3	12	83
	K1	w%	31.8	31.8	31.8	31.8	0.7	2.2	4	18	94

Rob. mean: the robust mean, s_{rob} : the robust standard deviation, $s_{rob} \%$: the robust standard deviation as percent, s : the standard deviation, $s \%$: the standard deviation as percent, $2 \times s_{pt} \%$: the standard deviation for proficiency assessment at the 95 % confidence level, Acc z %: the results (%), where $|z| \leq 2$, n_{all} : the number of the participants.

In this proficiency test the participants were requested to report replicate results for all measurements. The results of the replicate determinations based on the ANOVA statistics are presented in Table 2. The targets for the repeatability are the ones recommended in the international standards related to the measurements of fuels. In particular, in measurements of the calorific values, the requirement for the repeatability is ± 120 J/g

Table 2. The summary of repeatability on the basis of replicate determinations (ANOVA statistics).

Measurand	Sample	Unit	Assigned value	Mean	s_w	s_b	s_t	$s_w\%$	$s_b\%$	$s_t\%$	s_b/s_w
Ash _d	B1	w%	5.38	5.40	0.054	0.185	0.192	1.0	3.4	3.6	3.4
	B2	w%	0.27	0.28	0.016	0.039	0.042	6.0	14	16	2.4
	K1	w%	10.8	10.8	0.057	0.112	0.126	0.53	1.0	1.2	2.0
C _d	B1	w%	54.7	54.5	0.080	0.455	0.462	0.15	0.84	0.85	5.7
	B2	w%	50.2	50.3	0.392	1.49	1.54	0.79	3.0	3.1	3.8
	K1	w%	72.3	72.3	0.134	0.628	0.642	0.19	0.87	0.89	4.7
EF	B1	t CO ₂ /TJ	106	106	0.222	0.676	0.712	0.21	0.64	0.67	3.0
	K1	t CO ₂ /TJ	94.2	94.2	0.123	0.982	0.990	0.13	1.0	1.0	8.0
H _d	B1	w%	5.74	5.70	0.042	0.210	0.215	0.73	3.7	3.8	5.0
	B2	w%	5.93	5.88	0.072	0.230	0.241	1.2	3.9	4.1	3.2
	K1	w%	4.42	4.44	0.062	0.142	0.155	1.4	3.2	3.5	2.3
M _{ad,d}	B1	w%	9.10	9.10	0.055	0.337	0.342	0.60	3.7	3.8	6.2
	B2	w%	8.87	8.88	0.095	1.29	1.29	1.1	15	15	14
	K1	w%	3.47	3.47	0.049	0.277	0.281	1.4	8.0	8.1	5.7
N _d	B1	w%	2.71	2.73	0.027	0.078	0.083	1.0	2.9	3.0	2.9
	B2	w%	0.08	0.09	0.015	0.034	0.0371	16	36	39	2.3
	K1	w%	2.18	2.15	0.037	0.114	0.120	1.7	5.3	5.6	3.1
Q _{p,net,d}	B1	J/g	21254	21264	44.6	146	153	0.21	0.69	0.72	3.3
	B2	J/g	18821	18820	69.5	139	156	0.37	0.74	0.83	2.0
	K1	J/g	28203	28216	57.2	156	166	0.20	0.55	0.59	2.7
Q _{V,gr,d}	B1	J/g	22471	22466	38.2	118	124	0.17	0.52	0.55	3.1
	B2	J/g	20142	20136	111	160	194	0.55	0.79	0.97	1.4
	K1	J/g	29137	29137	68.0	101	121	0.23	0.35	0.42	1.5
S _d	B1	w%	0.20	0.20	0.005	0.014	0.015	2.6	7.0	7.5	2.7
	K1	w%	0.31	0.31	0.016	0.020	0.026	5.2	6.6	8.4	1.3
V _{db}	B1	w%	70.2	70.2	0.095	0.801	0.807	0.14	1.1	1.1	8.4
	B2	w%	85.2	85.1	0.212	1.29	1.31	0.25	1.5	1.5	6.1
	K1	w%	31.8	31.8	0.127	0.701	0.713	0.40	2.2	2.2	5.5

Ass.val.: assigned value; s_w : repeatability standard error; s_b : between participants standard error; s_t : reproducibility standard error.

In this proficiency test the requirements for the repeatability of the measurements of the gross calorific value were 0.53 % for the sample B1, 0.60 % for the sample B2 and 0.41 % for the sample K1 and in measurement of the net calorific value 0.56 %, 0.64 % and 0.43 %, respectively. In each case, the obtained repeatability of the measurement of the gross calorific value and the net calorific value was lower than the repeatability requirement (Table 2, the column s_w %).

The estimation of the robustness of the methods could be done by the ratio s_b/s_w . The ratio s_b/s_w should not exceed the value 3 for robust methods. Here, however, the robustness exceeded the value 3 in many cases (Table 2). For the gross calorific value, the ratio s_b/s_w , was 3.1 (the sample B1), 1.4 (the sample B2) and 1.5 (the sample K1), for the net calorific values 3.3, 2.0 and 2.7, respectively. For the calorific values the ratio s_b/s_w was mainly within the same range than in the previous similar proficiency tests CAL 05/2009 (B1), CAL 05/2010 (K1), and CAL 07/2018, but mainly the values were lower than the previous ones [4, 5, 7].

3.2 Analytical methods

The participants were allowed to use different analytical methods for the measurands in the PT. A survey of the used analytical methods was carried out along the proficiency test. The summary of the survey is shown in Appendix 11. The used analytical methods and the results of the participants grouped by methods are shown in more detail in Appendix 12. The statistical comparison of the analytical methods was possible for the data where the number of the results was ≥ 5 (several cases in this PT). In those cases the comparison is based on the graphical result evaluation.

3.2.1 Gross and net calorific value

The analytical methods based on different standard methods were used for the measurements in this PT. The used analytical methods of the participants are shown in more detail in Appendices 11 and 12.

Mostly standard methods were used for measurement of calorific values ($q_{v,gr,d}$ and $q_{p,net,d}$) (EN 14918 [8], EN ISO 18125 [9], ISO 1928 [10], Appendix 12). One to two participants used standard ASTM D 5865 [11] or DIN 51900 [12]. Two participants (8, 15) used other standard method (EN 15400).

For the calculations of gross calorific value ($q_{v,gr,d}$), various correction factors were used. Fuse wire, ignition, acid, moisture, nitrogen and sulphur corrections were most commonly used in several different combinations depending of the test material (Appendix 11). Also for the calculations of net calorific value ($q_{p,net,d}$), different combinations of correction factors were used well depending of the test material (Appendix 11). Mainly nitrogen plus oxygen (N+O) and hydrogen (H) content was used for corrections. Based on the statistical comparison and the graphical evaluation no clear differences between the used methods in gross and net calorific value measurements could be concluded (Appendix 12).

3.2.2 Measurement of ash, carbon, hydrogen, moisture, nitrogen, sulphur, and volatile matter

In the PT mainly the following standard methods or technical specifications were used for measurements of different parameters (Appendix 12):

Measurand	Method
Ash _d	EN 14775 [13], ISO 1171 [14], EN ISO 18122 [15], ASTM D 7582 [16]
C _d , H _d and N _d	ISO 29541 [17], ASTM D 5373 [18], EN ISO 16948 [19]
M _{ad} (analytical moisture content)	EN 14774-3 [20], ISO 589 [21], DIN 51718 [22], ASTM D 7582 [16], EN ISO 18134-3 [23], ISO 11722 [24]
S _d	ISO 334 [25], EN ISO 16994 [26], ASTM D 4239 [27]
V _{db} , (volatile matter)	EN 15148 [28], ISO 562 [29], EN ISO 18123 [30]

However, in some cases also other international and national standards or technical specifications (e.g. EN 15403, ASTM D 4422, EN 13137, EN 15407, ISO 1928, ISO 333, EN 15934, ASTM D 3173, EN 15414, ISO 5068, ASTM D 6376, ISO 19579, EN 15402, ASTM D 3175, EN 15402, ASTM D 7582, ISO 5071) or internal methods were used.

The ash content was determined mainly gravimetrically by heating at the temperature 500 °C (Sample B2), at the temperature 550 °C (Samples B1 and B2), at the temperature 700, 710 or 815 °C (Sample K1) or at the temperature 815 °C (Sample B1). Ash content was measured also using TGA for the samples at the temperatures between 550 °C and 815 °C (Appendix 11). In the international standards EN 14775 and EN ISO 18122 the ashing temperature is mentioned to be 550 °C for solid biofuels [13, 15]. While in ISO 1711 for solid mineral fuels it is mentioned to be 815 °C [14]. Based on the graphical result evaluation, clear differences between the used methods in measurements could not be concluded (Appendix 12).

Moisture content was determined gravimetrically by heating in air or N₂ atmosphere at the temperatures of 105-108°C. Moisture content was measured also using TGA at the temperatures of 105-107 °C (Appendix 11).

Most of the participants conducted CHN analyses from air dried samples, one participant used dried B1 sample, two participants used dried B2 and K1 samples, and five participants dried K1 sample (Appendix 11). For C_d results a statistically significant difference was observed between the international standard methods ASTM D 5373 (mean ± standard deviation, 72.8 ± 0.8, n = 5) and ISO 29541 (72.1 ± 0.3, n = 8) in the coal sample K1 (Appendices 12, 13).

For S_d no statistically significant difference between the used analytical method was noticed. For V_{db} in the coal sample K1 a statistically significant difference was observed between the international standard ISO 562 (31.5 ± 0.5, n = 12) and the used other methods (32.4 ± 0.7, n = 6, Appendix 13).

In the PT also information of the detection limits for nitrogen and sulphur was collected. The detection limits varied greatly for N: 0.001-50 w% and for S: 0.001-15000 w% (Appendix 11). Possible the high maximum values are reported invalid values.

3.3 Uncertainties of the results

At maximum 80 % of the participants reported the expanded uncertainties ($k=2$) with their results for at least some of their results (Appendix 14). The range of the reported uncertainties varied between the measurements and the sample types (Table 3).

Several approaches were used for estimating of measurement uncertainty (Appendix 14). The most used approaches were based on IQC data and method validation data. One participant reported the usage of the MUKIT measurement uncertainty software for the estimation of their uncertainties [31]. The free software is available on the webpage: www.syke.fi/envical/en. Generally, the used approach for estimating measurement uncertainty did not make definite impact on the uncertainty estimates.

The estimated uncertainties varied highly for all the tested measurands (Table 3). Especially, very low or high uncertainties can be considered questionable. It was evident, that **some uncertainties had been reported erroneously** for the measurands (including calorific values, Appendix 14), **not as relative values (%) as the provider of this proficiency test had requested**. It is evident, that harmonization is still needed for the estimation of the expanded measurement uncertainties.

Table 3. The range of the expanded measurement uncertainties ($k=2$, $U_i\%$) reported by the participants.

Measurement	Uncertainty B1, %	Uncertainty B2, %	Uncertainty K1, %
Ash _d	4.8-20	5-44.4	0.1-20
C _d	0.6-3.77	0.67-40	0.27-10
EF	4-10	-	2-6
H _d	5-14.6	0.55-20	0.25-20
M _{ad,d}	0.84-20	0.68-25.8	0.02-20
N _d	6-17	4.54-40	0.15-20
q _{p,net,d}	0.18-4	0.18-140	0.12-151
q _{V,gr,d}	0.18-8	0.18-140	0.09-151
S _d	7.98-30	-	0.01-30
V _{db}	0.39-5	0.39-10	0.18-5.5

3.4 Estimation of emission factor

Additionally, the participants were asked to estimate the emission factors (EF) for the peat and coal samples distributed in the PT by taking into account their own net calorific values and the total moisture values as received, which was informed in the cover letter of the samples. The calculation of the emission factor of the wood pellet sample (B2) was not done as it is a CO₂ neutral fuel. In this PT, very few participants reported their results for the emission factor (4-7). Due to the low number of the results, the performance evaluation was not given for the emission factor.

4 Evaluation of the results

The performance evaluation was based on the z scores, which were interpreted as follows:

Criteria	Performance
$ z \leq 2$	Satisfactory
$2 < z < 3$	Questionable
$ z \geq 3$	Unsatisfactory

In total, 90 % of the results evaluated based on z scores were satisfactory when accepting the deviation of 1–30 % from the assigned value (Appendix 9). About 76 % of the participants used the accredited methods and 94 % of their results were satisfactory. In the previous similar proficiency test CAL 07/2018 the performance was satisfactory for 89 % of the results when deviation 1–30 % from the assigned value was accepted [7].

Table 4. Summary of the performance evaluation in the proficiency test CAL 07/2019.

Sample	Satisfactory results (%)	Accepted deviation from the assigned value (%)	Remarks
Peat, B1	93	1.4-15	<ul style="list-style-type: none"> • Very good performance. • In the CAL 07/2018 the performance was satisfactory for 95 % of the results, when accepting 1.3-20 % deviation from the assigned value [7].
Wood pellet, B2	89	1.4-30	<ul style="list-style-type: none"> • Good performance. • Difficulties in measurements for $q_{p,gr,d} < 80\%$ satisfactory results. • In the CAL 07/2018 the performance was satisfactory for 83 % of the results [7].
Coal, K1	92	1-15	<ul style="list-style-type: none"> • Very good performance. • In the CAL 07/2018 the performance was satisfactory for 89 % of the results [7].

The summary of the performance evaluation is shown in Table 4. The percentage of the satisfactory results varied between 89 % and 93 % for the tested sample types. The criteria for performance evaluation is mainly set according to the target value for reproducibility recommended in international standards or technical specifications for measurement of the calorific values and other determinants. The reproducibility required in the standards was fulfilled for the gross calorific values. For the net calorific value increased reproducibility from the value for the gross calorific value was used. There was no criterion for reproducibility for the net calorific value in standards methods.

Peat

In the previous similar PT (CAL 07/2018) 95 % of the results were satisfactory for the peat sample (B1) when accepting 1.3–30 % deviation from the assigned value [7], and thus the performance was in the same range in this PT (93 %, Table 4). The number of satisfactory results of the gross and net calorific values for peat sample was nearly the same for the gross calorific value and the net calorific value when compared to the previous similar PT [7]. The results of analysis moisture (M_{ad}) and emission factor (EF) have not been evaluated, but the assigned values are presented (Table 1).

Wood pellet

In the previous similar PT CAL 07/2018 the satisfactory results of the wood pellet sample (B2) were in total 83 %, when accepting deviation 1.4–30 % from the assigned value [7], thus the performance in this PT was slightly higher (89 %, Table 4). The satisfactory results varied between 76 % ($q_{p,gr,d}$) and 94 % (Ash_d) for the wood pellet sample (Table 1). In the measurement of gross and net calorific values 76 % and 88 % of the results, respectively, were satisfactory when accepting deviations of 1.4 % and 1.8 % from the assigned values (Table 1). The number of satisfactory results was lower for the gross calorific values and higher for the net calorific value for wood pellet than in the previous similar PT CAL 07/2018 (83 % and 73, respectively) [7]. The estimation of EF was not done as it is a CO₂ neutral fuel. Also, the results of analysis moisture (M_{ad}) and nitrogen (N_d) have not been evaluated, but the assigned value is given (Table 1).

Coal

In the previous similar PT CAL 07/2018 the satisfactory results of the coal sample (K1) were in total 89 % [5], thus the performance was higher in this PT (92 %, Table 4). In the measurement of gross and net calorific values, 86 % and 93 % of results, respectively, were satisfactory, when accepting the deviations of 1 and 1.3 % from the assigned values (Table 1). These were almost at the same level for the gross calorific value and higher for the net calorific value than in the previous similar PT CAL 07/2018 (88 % and 79 %, respectively) [7]. The results of analysis moisture (M_{ad}) and emission factor (EF) have not been evaluated, but the assigned value is given (Table 1).

5 Summary

Profest SYKE carried out the proficiency test (PT) for the analysis of the gross and the net calorific value as well as for content of ash, carbon, hydrogen, nitrogen, sulphur, analytical moisture content and volatile matter in fuels in August-September 2019. Three types of samples were delivered to the participants: peat, wood pellet (not sulphur) and coal. In total 34 participants took part in the PT. The participants also had the possibility to estimate or calculate the emission factor for peat and coal samples.

The robust mean or the median of the results reported by the participants were used as the assigned values for measurands. The uncertainty for the assigned value was estimated at the 95 % confidence level and it was less than 0.7 % for calorific values and at maximum 7.5 % for the other measurands.

The evaluation of the performance was based on the z scores, which were calculated using the standard deviation for proficiency assessment at 95 % confidence level. The evaluation of performance was not done for the measurement of M_{ad} in all samples, N_d in the wood pellet sample, and EF for peat and coal samples. In this proficiency test 90 % of the data was regarded to be satisfactory when, depending on the measurand and sample, the result was accepted to deviate from the assigned value from 1 to 30 %. About 76 % of the participants used the accredited methods and 94 % of their results were satisfactory. In measurements of the gross calorific value from the peat, the wood pellet and the coal samples, 93 %, 76 % and 86 % of the results were satisfactory, respectively. In measurements of the net calorific value from the peat, the wood pellet and the coal samples, 82 %, 88 % and 93 % of the results were satisfactory, respectively. In general, the results were in the same range as in the previous similar Profest SYKE proficiency test, CAL 07/2018 [7], but the performance in the gross calorific value was somewhat lower for wood pellet and higher for the net calorific value for wood pellet and coal samples in the present PT.

6 Summary in Finnish

Proftest SYKE järjesti elo-syyskuussa 2019 pätevyyskokeen kalorimetrisen ja tehollisen lämpöarvon sekä tuhkan, hiilen, vedyn, typen, rikin, kosteuden ja haihtuvien yhdisteiden määrittämiseksi turpeesta, puupelletistä (ei rikkiä) ja kivihiilestä. Pätevyyskokeeseen osallistui yhteensä 34 laboratoriota. Lisäksi osallistujilla oli mahdollisuus laskea päästökerron turve- ja kivihiilinäytteistä.

Testisuureen vertailuarvona käytettiin osallistujien ilmoittamien tulosten robustia keskiarvoa tai niiden mediaania. Vertailuarvon epävarmuus oli lämpöarvomäärittelyssä alhaisempi kuin 0,7 % ja muiden määritysten osalta korkeintaan 7,5 %.

Osallistujien pätevyys arviointi tehtiin z-arvojen avulla ja niiden laskemisessa käytetyt tavoitehajonnat olivat määrittämisestä ja näytteestä riippuen välillä 1–30 %. Tulosten arviointia ei tehty testinäytteiden kosteuspitoisuuden määrittämiselle, typen määrittämiselle puupelletistä eikä turpeen ja kivihiilen päästökertoimelle. Koko tulosaineistossa hyväksyttävää tuloksia oli 90 %, kun vertailuarvosta sallittiin 1–30 % poikkeama. Noin 76 % osallistujista käytti akkreditoituja määrittämenetelmiä ja näistä tuloksista oli hyväksyttävää 94 %. Kalorimetrisen lämpöarvon tuloksista oli hyväksyttävää 93 % (turve), 76 % (puupelletti) ja 86 % (kivihiili). Tehollisen lämpöarvon tuloksille vastaavat hyväksyttävien tulosten osuudet olivat 82 % (turve), 88 % (puupelletti) ja 93 % (kivihiili). Hyväksyttävää tuloksia oli lähes saman verran kuin edellisessä vastaavassa pätevyyskokeessa CAL 07/2018 [7]. Puupellettinäytteen osalta kalorimetrisen lämpöarvon menestyminen oli alhaisempi, mutta tehollisen lämpöarvon menestyminen oli parempi puupelletti- ja kivihiilinäytteen osalta kuin edellisellä kierroksella.

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APPENDIX 1: Participants in the proficiency test

Country	Participant
Bosnia-Hertsegovina	JP Elektroprivreda d.d.Sarajevo, Z.D. RMU Kakanj d.o.o Kakanj
Bulgary	AES-3C Maritza East 1 EOOD; Testing Laboratory "Energy Materials" Energy Agency of Plovdiv
Estonia	Enefit Energiatootmine AS Chemical Laboratory
Finland	Eurofins Labtium Oy, Jyväskylä Eurofins Nab Labs Oy, Naantali Finnsementti Oy Fortum Waste Solutions Oy, Riihimäki FTF Fuel Testing Finland Oy KVVY-Botnialab, Vaasa Kymen Ympäristölaboratorio Oy Kymenlaakson ammattikorkeakoulu Luonnonvarakeskus Kokkolan laboratorio SSAB Europe Raahе, Raahе SYNLAB Analytics & Services Finland Oy
France	ArcelorMittal Fos sur Mer CARSO CAE - Laboratoire de Toulouse Eurofins Analyses des Matériaux et Combustibles France SOCOR Dechy France
Germany	GBA Gesellschaft für Bioanalytik mbH
Hungary	Dunafer Labor Nonprofit Kft. Szénkémiai A. Foosztály
Lithuania	AB "Siaulių Energija" chemijos laboratorija, Siauliai, Lithuania Orion Global PET
Republic of Ireland	Edenderry Power Ltd
Republic of Korea	Komipo, Boryeong Thermal Power Site Division Korea Conformity Laboratories (KCL)
Romania	CRH Cement (Romania)-Punct de lucru Hoghiz Holcim Romania -Ciment Alesd Laborator analize fizico-chimice apa si carbune, Romania Romp petrol Quality Control SRL-Laborator Produse Petroliere
Slovenia	Salonit Anhovo
Spain	Centro de Investigacion Elias Masaveu S.A. Laboratorio Central de Calidad - LCC
Sweden	RISE Research Institutes of Sweden AB

APPENDIX 2: Preparation of the samples

Sample B1, peat

The peat sample B1 was reused material from an earlier PT CAL 05/2009 and the sample preparation is described more detail in the final report of that PT [4].

Sample B2, wood pellet

Sample B2 was prepared from spruce sawdust. The wood pellets were first crushed with a cutting mill and then ground by the mill with 1000 µm sieve at the laboratory of Eurofins Labtium Ltd. The sieved sample was mixed by a mechanized sample mixer and distributed to subsamples of ca. 30 g using a rotary sample divider equipped with a vibratory sample feeder at the laboratory of KVVY Tutkimus Oy (Tampere).

Sample K1

The coal sample K1 was reused material from an earlier PT CAL 05/2010 and the sample preparation is described more detail in the final report of that PT [5].

APPENDIX 3: Homogeneity of the samples

Homogeneity was tested from duplicate measurements of calorific value (Table 1) and ash content in five samples from the sample B2 and in three samples from the samples B1 and K1, which were homogenised before sampling. Additionally, the other measurands from two samples was tested.

Criteria for homogeneity:

$s_{anal}/s_h < 0.5$ and $s_{sam}^2 < c$, where

s_h % = standard deviation for testing of homogeneity

s_{anal} = analytical deviation, standard deviation of the results within sub samples

s_{pt} % = standard deviation for proficiency assessment

s_{sam} = between-sample deviation, standard deviation of the results between sub samples

$c = F1 \times s_{all}^2 + F2 \times s_{anal}^2$, where
 $s_{all}^2 = (0.3 \times s_h)^2$,

F1 and F2 are constants of F distribution derived from the standard statistical tables for the tested number of samples [2, 3].

Table 1. Results from the homogeneity testing of the peat (B1), pellet (B2) and coal (K1) samples.

Measurements	n	Mean	s_h %	s_{pt} %	s_h	s_{anal}	s_{anal}/s_h	Is $s_{anal}/s_h < 0.5$?	s_{sam}	s_{sam}^2	c	Is $s_{sam}^2 < c$?
Peat (B1)												
Gross calorific value, J/g	3	22528	0.3	0.7	158	24.2	0.15	yes	26.1	680	9210	yes
Net calorific value, J/g	3	21253	0.3	0.75	159	24.5	0.15	yes	26.3	690	9410	yes
Pellet (B2)												
Gross calorific value, J/g	5	20312	0.7	0.7	142	68.1	0.48	yes	66.2	4380	13450	yes
Net calorific value, J/g	5	19046	0.8	0.9	171	67.6	0.39	yes	65.8	4330	15480	yes
Coal (K1)												
Gross calorific value, J/g	3	29230	0.2	0.5	146	24.6	0.17	yes	0	0	8340	yes
Net calorific value, J/g	3	28292	0.2	0.65	184	24.6	0.13	yes	0	0	11700	yes

n = number of tested samples

Conclusion: In each case, the criteria were fulfilled. **Thus, all the samples could be regarded as homogenous.** Also the results of the other tested measurands confirm the homogeneity of the samples.

APPENDIX 4: Feedback from the proficiency test

FEEDBACK FROM THE PARTICIPANTS

Participant	Comments on technical execution	Action / Profest SYKE
8	The participant informed receiving the samples on 13 th September.	According to the distributor's (Posti) tracking system the samples arrived to the pick-up location on 30 th August. The provider recommends to check the internal package delivery procedures.

Participant	Comments to the results	Action / Profest SYKE
16, 25, 30	The participant did not deliver the results to Profest SYKE by selecting "Send results" on ProfestWEB.	The provider accepted the results.
22	The participant reported erroneously their results of C_d in the wood pellet sample B2. Their correct values were: B2: 52.05 w%, 52.09 w%	The provider does not correct the results after delivering the preliminary results. The erroneous results were handled as outliers in the statistical treatment. They did not affect to the assigned value evaluation. If the C_d value had been reported correctly they would have been unsatisfactory. The participant can re-calculate the z scores according to the Guide for participants [6].
4	The participants asked the clarification of the non-evaluated performance of the emission factor in the sample K1.	The provider clarified the reasons for the non-evaluation and given proposal for the performance evaluation by the participant for their result.

FEEDBACK TO THE PARTICIPANTS

Participant	Comments
33	The participant reported only one result (Ash_d) instead of replicate results for some measurands. The results have been excluded from the calculation of the assigned values, and results are not evaluated. The participants should follow more carefully the instructions given by the provider.
23	The participant didn't report the method for Ash_d , H_d , Mad_d and N_d . The participants should follow more carefully the instructions given by the provider. The participant reported the use of external accredited laboratory for the measurement H_d and N_d . In the proficiency test the performance of the participating laboratory is evaluated, thus subcontracting is not allowable. The subcontracted results were eliminated from the database of the final report.
5, 6, 9, 17, 23	The participants did not report the expanded measurement uncertainties for some measurands. Participants are accredited laboratories, whom should report uncertainties with their results.
4, 5, 6, 7, 8, 6, 17, 33	For these participants the deviation of replicate measurements for some measurands and samples was high and their results were Cochran outliers. The provider recommends the participants to validate their accepted deviation of replicate measurements.
4, 7, 8, 19, 27, 32	It was evident, that some uncertainties had been reported erroneously for the measurands (including calorific values), not as relative values as the provider of this proficiency test had requested. The provider recommends the participants to follow more carefully the instructions given by the provider.

APPENDIX 5: Evaluation of the assigned values and their uncertainties

Measurand	Sample	Unit	Assigned value	U_{pt}	$U_{pt}, \%$	Evaluation method of assigned value	u_{pt}/s_{pt}
Ash _d	B1	w%	5.38	0.11	2.1	Robust mean	0.30
	B2	w%	0.27	0.02	7.5	Robust mean	0.25
	K1	w%	10.8	0.0	0.3	Robust mean	0.12
C _d	B1	w%	54.7	0.4	0.7	Median	0.28
	B2	w%	50.2	0.4	0.7	Median	0.28
	K1	w%	72.3	0.4	0.6	Robust mean	0.24
EF	B1	t CO ₂ /TJ	106	1	0.7	Median	-
	K1	t CO ₂ /TJ	94.2	0.2	0.2	Median	-
H _d	B1	w%	5.74	0.17	3.0	Median	0.33
	B2	w%	5.93	0.16	2.7	Median	0.27
	K1	w%	4.42	0.09	2.0	Robust mean	0.29
M _{ad,d}	B1	w%	9.10	0.25	2.7	Robust mean	-
	B2	w%	8.87	0.13	1.5	Robust mean	-
	K1	w%	3.47	0.09	2.7	Robust mean	-
N _d	B1	w%	2.71	0.07	2.4	Median	0.24
	B2	w%	0.08	-	-	Median	-
	K1	w%	2.18	0.08	3.6	Median	0.36
q _{p,net,d}	B1	J/g	21254	106	0.5	Median	0.33
	B2	J/g	18821	113	0.6	Robust mean	0.33
	K1	J/g	28203	85	0.3	Robust mean	0.23
q _{V,gr,d}	B1	J/g	22471	67	0.3	Robust mean	0.21
	B2	J/g	20142	81	0.4	Robust mean	0.29
	K1	J/g	29137	58	0.2	Robust mean	0.20
S _d	B1	w%	0.20	0.01	4.8	Median	0.32
	K1	w%	0.31	0.01	3.1	Robust mean	0.21
V _{ab}	B1	w%	70.2	0.6	0.9	Median	0.30
	B2	w%	85.2	0.8	0.9	Median	0.30
	K1	w%	31.8	0.4	1.3	Robust mean	0.33

U_{pt} = Expanded uncertainty of the assigned value

Criterion for reliability of the assigned value $u_{pt}/s_{pt} \leq 0.3$, where

s_{pt} = the standard deviation for proficiency assessment

u_{pt} = the standard uncertainty of the assigned value

If $u_{pt}/s_{pt} \leq 0.3$, the assigned value is reliable and the z scores are qualified.

APPENDIX 6: Terms in the results tables

Results of each participant

Measurand	The tested parameter
Sample	The code of the sample
z score	Calculated as follows: $z = (x_i - x_{pt})/s_{pt}$, where x_i = the result of the individual participant x_{pt} = the assigned value s_{pt} = the standard deviation for proficiency assessment
Assigned value	The value attributed to a particular property of a proficiency test item
$2 \times s_{pt}$ %	The standard deviation for proficiency assessment (s_{pt}) at the 95 % confidence level
Participant's result	The result reported by the participant (the mean value of the replicates)
Md	Median
s	Standard deviation
s %	Standard deviation, %
n_{stat}	Number of results in statistical processing

Summary on the z scores

S – satisfactory ($-2 \leq z \leq 2$)

Q – questionable ($2 < z < 3$), positive error, the result deviates more than $2 \times s_{pt}$ from the assigned value

q – questionable ($-3 < z < -2$), negative error, the result deviates more than $2 \times s_{pt}$ from the assigned value

U – unsatisfactory ($z \geq 3$), positive error, the result deviates more than $3 \times s_{pt}$ from the assigned value

u – unsatisfactory ($z \leq -3$), negative error, the result deviates more than $3 \times s_{pt}$ from the assigned value

Robust analysis

The items of data are sorted into increasing order, $x_1, x_2, x_i, \dots, x_p$.

Initial values for x^* and s^* are calculated as:

$$x^* = \text{median of } x_i (i = 1, 2, \dots, p)$$

$$s^* = 1.483 \times \text{median of } |x_i - x^*| (i = 1, 2, \dots, p)$$

The mean x^* and s^* are updated as follows:

Calculate $\varphi = 1.5 \times s^*$. A new value is then calculated for each result x_i ($i = 1, 2, \dots, p$):

$$x_i^* = \begin{cases} x^* - \varphi, & \text{if } x_i < x^* - \varphi \\ x^* + \varphi, & \text{if } x_i > x^* + \varphi, \\ x_i & \text{otherwise} \end{cases}$$

The new values of x^* and s^* are calculated from:

$$x^* = \sum x_i^* / p$$

$$s^* = 1.134 \sqrt{\sum (x_i^* - x^*)^2 / (p-1)}$$

The robust estimates x^* and s^* can be derived by an iterative calculation, i.e. by updating the values of x^* and s^* several times, until the process convergences [2].

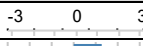








APPENDIX 7: Results of each participant

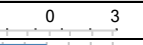




Participant 1												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
C _d	w%	B2		-1.35	50.2	2.5	49.4	50.2	50.3	0.5	0.9	8
M _{ad,d}	w%	B2			8.87		8.96	8.87	8.88	0.22	2.4	22
Q _{V,gr,d}	J/g	B2		-142.71	20142	1.4	20	20167	20136	178	0.9	19



























Participant 2												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
M _{ad,d}	w%	B1			9.10		9.27	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.92	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.31	3.45	3.47	0.17	5.0	24
Q _{p,net,d}	J/g	B1		-18.27	21254	1.5	18342	21254	21264	149	0.7	9
	J/g	B2		-15.75	18821	1.8	16154	18821	18820	148	0.8	15
Q _{V,gr,d}	J/g	B1		-1.77	22471	1.4	22193	22484	22466	121	0.5	13
	J/g	B2		2.78	20142	1.4	20534	20167	20136	178	0.9	19
	J/g	K1		-4.23	29137	1	28521	29139	29137	111	0.4	19

Participant 3												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		-1.06	5.38	7	5.18	5.45	5.40	0.13	2.4	13
	w%	B2		-0.12	0.27	30	0.27	0.29	0.28	0.03	9.2	18
M _{ad,d}	w%	B1			9.10		9.26	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.80	8.87	8.88	0.22	2.4	22

Participant 4												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		0.37	5.38	7	5.45	5.45	5.40	0.13	2.4	13
	w%	B2		-0.74	0.27	30	0.24	0.29	0.28	0.03	9.2	18
	w%	K1		2.33	10.8	2.5	11.1	10.8	10.8	0.0	0.4	21
C _d	w%	B1		0.20	54.7	2.5	54.8	54.7	54.5	0.5	0.8	6
	w%	B2		0.91	50.2	2.5	50.8	50.2	50.3	0.5	0.9	8
	w%	K1		-0.43	72.3	2.5	71.9	72.2	72.3	0.6	0.9	15
EF	t CO ₂ /TJ	B1			106		106	106	106	1	0.7	4
	t CO ₂ /TJ	K1			94.2		94.2	94.2	94.2	0.2	0.2	5
H _d	w%	B1		-1.65	5.74	9	5.31	5.74	5.70	0.21	3.7	6
	w%	B2		0.62	5.93	10	6.11	5.93	5.88	0.24	4.0	9
	w%	K1		1.44	4.42	7	4.64	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	B1			9.10		9.40	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		9.12	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.41	3.45	3.47	0.17	5.0	24
N _d	w%	B1		-0.06	2.71	10	2.70	2.71	2.73	0.08	2.9	6
	w%	B2			0.08		0.07	0.08	0.09	0.04	37.4	7
	w%	K1		-1.25	2.18	10	2.04	2.18	2.15	0.12	5.5	9
Q _{p,net,d}	J/g	B1		1.67	21254	1.5	21520	21254	21264	149	0.7	9
	J/g	B2		1.02	18821	1.8	18994	18821	18820	148	0.8	15
	J/g	K1		0.46	28203	1.3	28288	28212	28216	161	0.6	15

Participant 4													
Measurand	Unit	Sample		z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
q _{V,gr,d}	J/g	B1		1.32	22471	1.4	22679	22484	22466	121	0.5	13	
	J/g	B2		1.29	20142	1.4	20324	20167	20136	178	0.9	19	
	J/g	K1		1.06	29137	1	29291	29139	29137	111	0.4	19	
S _d	w%	B1		-0.27	0.20	15	0.20	0.20	0.20	0.01	7.2	9	
	w%	K1		-1.68	0.31	15	0.27	0.31	0.31	0.01	3.8	18	
V _{db}	w%	B1		0.26	70.2	3	70.5	70.2	70.2	0.8	1.1	7	
	w%	B2		0.39	85.2	3	85.7	85.2	85.1	1.3	1.5	11	
	w%	K1		-0.11	31.8	4	31.7	31.8	31.8	0.7	2.2	18	

Participant 5													
Measurand	Unit	Sample		z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
q _{p,net,d}	J/g	B1		-4.18	21254	1.5	20588	21254	21264	149	0.7	9	
	J/g	B2		-1.10	18821	1.8	18635	18821	18820	148	0.8	15	
q _{V,gr,d}	J/g	B1		-3.04	22471	1.4	21994	22484	22466	121	0.5	13	
	J/g	B2		-0.29	20142	1.4	20101	20167	20136	178	0.9	19	

Participant 6													
Measurand	Unit	Sample		z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{sta}	
Ash _d	w%	B1		-1.01	5.38	7	5.19	5.45	5.40	0.13	2.4	13	
	w%	B2		0.86	0.27	30	0.31	0.29	0.28	0.03	9.2	18	
	w%	K1		-0.07	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21	
C _d	w%	B1		0.20	54.7	2.5	54.8	54.7	54.5	0.5	0.8	6	
	w%	B2		0.89	50.2	2.5	50.8	50.2	50.3	0.5	0.9	8	
	w%	K1		-0.61	72.3	2.5	71.8	72.2	72.3	0.6	0.9	15	
EF	t CO ₂ /TJ	B1			106		107	106	106	1	0.7	4	
	t CO ₂ /TJ	K1			94.2		93.9	94.2	94.2	0.2	0.2	5	
H _d	w%	B1		-0.02	5.74	9	5.74	5.74	5.70	0.21	3.7	6	
	w%	B2		-0.02	5.93	10	5.93	5.93	5.88	0.24	4.0	9	
	w%	K1		-2.36	4.42	7	4.06	4.39	4.44	0.10	2.4	12	
M _{ad,d}	w%	B1			9.10		8.96	9.11	9.10	0.34	3.7	15	
	w%	B2			8.87		8.85	8.87	8.88	0.22	2.4	22	
	w%	K1			3.47		3.56	3.45	3.47	0.17	5.0	24	
N _d	w%	B1		-0.12	2.71	10	2.69	2.71	2.73	0.08	2.9	6	
	w%	B2			0.08		0.06	0.08	0.09	0.04	37.4	7	
	w%	K1		-2.44	2.18	10	1.91	2.18	2.15	0.12	5.5	9	
q _{p,net,d}	J/g	B1		0.09	21254	1.5	21269	21254	21264	149	0.7	9	
	J/g	B2		0.71	18821	1.8	18941	18821	18820	148	0.8	15	
	J/g	K1		0.58	28203	1.3	28310	28212	28216	161	0.6	15	
q _{V,gr,d}	J/g	B1		0.25	22471	1.4	22511	22484	22466	121	0.5	13	
	J/g	B2		0.63	20142	1.4	20232	20167	20136	178	0.9	19	
	J/g	K1		0.29	29137	1	29179	29139	29137	111	0.4	19	
S _d	w%	B1		0.07	0.20	15	0.20	0.20	0.20	0.01	7.2	9	
	w%	K1		-0.19	0.31	15	0.31	0.31	0.31	0.01	3.8	18	

Participant 7												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
C _d	w%	K1		-0.58	72.3	2.5	71.8	72.2	72.3	0.6	0.9	15
H _d	w%	K1		-0.32	4.42	7	4.37	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	K1			3.47		3.01	3.45	3.47	0.17	5.0	24
N _d	w%	K1		0.50	2.18	10	2.24	2.18	2.15	0.12	5.5	9
S _d	w%	K1		0.65	0.31	15	0.33	0.31	0.31	0.01	3.8	18

Participant 8												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B2		0.00	0.27	30	0.27	0.29	0.28	0.03	9.2	18
	w%	K1		1.85	10.8	2.5	11.1	10.8	10.8	0.0	0.4	21
C _d	w%	B2		-1.51	50.2	2.5	49.3	50.2	50.3	0.5	0.9	8
	w%	K1		0.91	72.3	2.5	73.1	72.2	72.3	0.6	0.9	15
H _d	w%	B2		-1.28	5.93	10	5.55	5.93	5.88	0.24	4.0	9
	w%	K1		0.73	4.42	7	4.53	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	B2			8.87		3.85	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.72	3.45	3.47	0.17	5.0	24
N _d	w%	B2			0.08		0.08	0.08	0.09	0.04	37.4	7
	w%	K1		0.62	2.18	10	2.25	2.18	2.15	0.12	5.5	9
q _{p,net,d}	J/g	B2		-4.68	18821	1.8	18029	18821	18820	148	0.8	15
	J/g	K1		0.16	28203	1.3	28232	28212	28216	161	0.6	15
q _{V,gr,d}	J/g	B2		-6.87	20142	1.4	19174	20167	20136	178	0.9	19
	J/g	K1		0.20	29137	1	29167	29139	29137	111	0.4	19
S _d	w%	K1		1.01	0.31	15	0.33	0.31	0.31	0.01	3.8	18
V _{db}	w%	B2		1.15	85.2	3	86.7	85.2	85.1	1.3	1.5	11
	w%	K1		1.16	31.8	4	32.5	31.8	31.8	0.7	2.2	18

Participant 9												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
q _{V,gr,d}	J/g	K1		0.36	29137	1	29190	29139	29137	111	0.4	19

Participant 10												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		-0.30	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	K1		1.28	72.3	2.5	73.5	72.2	72.3	0.6	0.9	15
M _{ad,d}	w%	K1			3.47		3.19	3.45	3.47	0.17	5.0	24
q _{p,net,d}	J/g	K1		-1.06	28203	1.3	28010	28212	28216	161	0.6	15
q _{V,gr,d}	J/g	K1		-0.38	29137	1	29082	29139	29137	111	0.4	19

Participant 11												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		-0.40	5.38	7	5.31	5.45	5.40	0.13	2.4	13
	w%	B2		-1.11	0.27	30	0.23	0.29	0.28	0.03	9.2	18
	w%	K1		0.04	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
M _{ad,d}	w%	B1			9.10		9.34	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.57	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		2.83	3.45	3.47	0.17	5.0	24
S _d	w%	B1		-1.43	0.20	15	0.18	0.20	0.20	0.01	7.2	9
	w%	K1		-0.15	0.31	15	0.31	0.31	0.31	0.01	3.8	18

Participant 11												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
V _{db}	w%	B1		0.02	70.2	3	70.2	70.2	70.2	0.8	1.1	7
	w%	B2		0.47	85.2	3	85.8	85.2	85.1	1.3	1.5	11
	w%	K1		0.60	31.8	4	32.2	31.8	31.8	0.7	2.2	18

Participant 12												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		-0.30	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	K1		-0.84	72.3	2.5	71.5	72.2	72.3	0.6	0.9	15
EF	t CO ₂ /TJ	K1			94.2		83.1	94.2	94.2	0.2	0.2	5
H _d	w%	K1		0.93	4.42	7	4.56	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	K1			3.47		4.33	3.45	3.47	0.17	5.0	24
N _d	w%	K1		0.00	2.18	10	2.18	2.18	2.15	0.12	5.5	9
q _{p,net,d}	J/g	K1		-0.78	28203	1.3	28060	28212	28216	161	0.6	15
q _{V,gr,d}	J/g	K1		-0.68	29137	1	29038	29139	29137	111	0.4	19
S _d	w%	K1		-0.69	0.31	15	0.29	0.31	0.31	0.01	3.8	18
V _{db}	w%	K1		0.12	31.8	4	31.9	31.8	31.8	0.7	2.2	18

Participant 13												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		0.69	5.38	7	5.51	5.45	5.40	0.13	2.4	13
	w%	B2		1.20	0.27	30	0.32	0.29	0.28	0.03	9.2	18
	w%	K1		1.44	10.8	2.5	11.0	10.8	10.8	0.0	0.4	21
C _d	w%	B1		-1.46	54.7	2.5	53.7	54.7	54.5	0.5	0.8	6
	w%	B2		-0.18	50.2	2.5	50.1	50.2	50.3	0.5	0.9	8
	w%	K1		-0.41	72.3	2.5	71.9	72.2	72.3	0.6	0.9	15
EF	t CO ₂ /TJ	B1			106		106	106	106	1	0.7	4
	t CO ₂ /TJ	K1			94.2		94.3	94.2	94.2	0.2	0.2	5
H _d	w%	B1		0.03	5.74	9	5.75	5.74	5.70	0.21	3.7	6
	w%	B2		-0.16	5.93	10	5.88	5.93	5.88	0.24	4.0	9
	w%	K1		-0.78	4.42	7	4.30	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	B1			9.10		8.89	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.73	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.62	3.45	3.47	0.17	5.0	24
N _d	w%	B1		0.11	2.71	10	2.72	2.71	2.73	0.08	2.9	6
	w%	B2			0.08		0.11	0.08	0.09	0.04	37.4	7
	w%	K1		0.74	2.18	10	2.26	2.18	2.15	0.12	5.5	9
q _{p,net,d}	J/g	B1		-0.57	21254	1.5	21163	21254	21264	149	0.7	9
	J/g	B2		-0.14	18821	1.8	18797	18821	18820	148	0.8	15
	J/g	K1		0.23	28203	1.3	28245	28212	28216	161	0.6	15
q _{V,gr,d}	J/g	B1		-0.39	22471	1.4	22410	22484	22466	121	0.5	13
	J/g	B2		-0.43	20142	1.4	20081	20167	20136	178	0.9	19
	J/g	K1		0.21	29137	1	29167	29139	29137	111	0.4	19
S _d	w%	B1		1.13	0.20	15	0.22	0.20	0.20	0.01	7.2	9
	w%	K1		0.00	0.31	15	0.31	0.31	0.31	0.01	3.8	18
V _{db}	w%	B1		0.00	70.2	3	70.2	70.2	70.2	0.8	1.1	7
	w%	B2		-0.04	85.2	3	85.2	85.2	85.1	1.3	1.5	11
	w%	K1		-0.36	31.8	4	31.6	31.8	31.8	0.7	2.2	18

APPENDIX 7 (5/10)

Participant 14												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		-0.30	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	K1		0.76	72.3	2.5	73.0	72.2	72.3	0.6	0.9	15
M _{ad,d}	w%	K1			3.47		3.40	3.45	3.47	0.17	5.0	24
S _d	w%	K1		-0.06	0.31	15	0.31	0.31	0.31	0.01	3.8	18
V _{db}	w%	K1		2.76	31.8	4	33.6	31.8	31.8	0.7	2.2	18

Participant 15												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		-2.66	5.38	7	4.88	5.45	5.40	0.13	2.4	13
	w%	B2		-0.25	0.27	30	0.26	0.29	0.28	0.03	9.2	18
	w%	K1		-0.33	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
M _{ad,d}	w%	B1			9.10		8.81	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.73	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.38	3.45	3.47	0.17	5.0	24
qV _{gr,d}	J/g	B1		-0.96	22471	1.4	22320	22484	22466	121	0.5	13
	J/g	B2		-2.92	20142	1.4	19731	20167	20136	178	0.9	19
	J/g	K1		-0.15	29137	1	29115	29139	29137	111	0.4	19
S _d	w%	B1		16.03	0.20	15	0.44	0.20	0.20	0.01	7.2	9
	w%	K1		6.71	0.31	15	0.47	0.31	0.31	0.01	3.8	18
V _{db}	w%	B1		8.55	70.2	3	79.2	70.2	70.2	0.8	1.1	7
	w%	B2		4.08	85.2	3	90.4	85.2	85.1	1.3	1.5	11
	w%	K1		0.83	31.8	4	32.3	31.8	31.8	0.7	2.2	18

Participant 16												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		0.44	10.8	2.5	10.9	10.8	10.8	0.0	0.4	21
M _{ad,d}	w%	K1			3.47		3.45	3.45	3.47	0.17	5.0	24
qV _{gr,d}	J/g	K1		-1.28	29137	1	28950	29139	29137	111	0.4	19
V _{db}	w%	K1		0.16	31.8	4	31.9	31.8	31.8	0.7	2.2	18

Participant 17												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		0.37	5.38	7	5.45	5.45	5.40	0.13	2.4	13
	w%	B2		0.62	0.27	30	0.30	0.29	0.28	0.03	9.2	18
	w%	K1		0.37	10.8	2.5	10.9	10.8	10.8	0.0	0.4	21
C _d	w%	B1		-0.15	54.7	2.5	54.6	54.7	54.5	0.5	0.8	6
	w%	B2		0.64	50.2	2.5	50.6	50.2	50.3	0.5	0.9	8
	w%	K1		0.00	72.3	2.5	72.3	72.2	72.3	0.6	0.9	15
H _d	w%	B1		0.66	5.74	9	5.91	5.74	5.70	0.21	3.7	6
	w%	B2		0.10	5.93	10	5.96	5.93	5.88	0.24	4.0	9
	w%	K1		-0.26	4.42	7	4.38	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	B1			9.10		9.13	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.91	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.62	3.45	3.47	0.17	5.0	24
N _d	w%	B1		-0.66	2.71	10	2.62	2.71	2.73	0.08	2.9	6
	w%	B2			0.08		0.16	0.08	0.09	0.04	37.4	7
	w%	K1		-1.10	2.18	10	2.06	2.18	2.15	0.12	5.5	9

Participant 17														
Measurand	Unit	Sample	<div><div>-303</div><div></div></div>	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}		
Q _{p.net.d}	J/g	B1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div>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Participant 18													
Measurand	Unit	Sample	<div><div><div>-3</div><div>0</div><div>3</div></div></div>	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
Ash 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Participant 21												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		1.01	5.38	7	5.57	5.45	5.40	0.13	2.4	13
	w%	K1		-0.28	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	B1		0.07	54.7	2.5	54.8	54.7	54.5	0.5	0.8	6
	w%	K1		0.12	72.3	2.5	72.4	72.2	72.3	0.6	0.9	15
H _d	w%	B1		-0.37	5.74	9	5.65	5.74	5.70	0.21	3.7	6
	w%	K1		-0.31	4.42	7	4.37	4.39	4.44	0.10	2.4	12
M _{ad,d}	w%	B1			9.10		9.11	9.11	9.10	0.34	3.7	15
	w%	K1			3.47		3.82	3.45	3.47	0.17	5.0	24
N _d	w%	B1		0.52	2.71	10	2.78	2.71	2.73	0.08	2.9	6
Q _{p,net,d}	J/g	B1		0.54	21254	1.5	21340	21254	21264	149	0.7	9
	J/g	K1		1.37	28203	1.3	28455	28212	28216	161	0.6	15
Q _{V,gr,d}	J/g	B1		0.62	22471	1.4	22568	22484	22466	121	0.5	13
	J/g	K1		1.88	29137	1	29412	29139	29137	111	0.4	19
S _d	w%	B1		-0.40	0.20	15	0.19	0.20	0.20	0.01	7.2	9
	w%	K1		0.13	0.31	15	0.31	0.31	0.31	0.01	3.8	18
V _{db}	w%	K1		0.87	31.8	4	32.4	31.8	31.8	0.7	2.2	18











Participant 22												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B2		-2.59	0.27	30	0.17	0.29	0.28	0.03	9.2	18
C _d	w%	B2		-7.24	50.2	2.5	45.7	50.2	50.3	0.5	0.9	8
H _d	w%	B2		-1.62	5.93	10	5.45	5.93	5.88	0.24	4.0	9
M _{ad,d}	w%	B2			8.87		11.84	8.87	8.88	0.22	2.4	22
N _d	w%	B2			0.08		0.12	0.08	0.09	0.04	37.4	7
Q _{p,net,d}	J/g	B2		1.51	18821	1.8	19076	18821	18820	148	0.8	15
Q _{V,gr,d}	J/g	B2		0.85	20142	1.4	20262	20167	20136	178	0.9	19
V _{db}	w%	B2		-2.33	85.2	3	82.2	85.2	85.1	1.3	1.5	11

Participant 23												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		0.66	5.38	7	5.51	5.45	5.40	0.13	2.4	13
	w%	B2		-0.25	0.27	30	0.26	0.29	0.28	0.03	9.2	18
	w%	K1		-0.07	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
M _{ad,d}	w%	B1			9.10		8.56	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.88	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.52	3.45	3.47	0.17	5.0	24
Q _{p,net,d}	J/g	B1		-1.66	21254	1.5	20990	21254	21264	149	0.7	9
	J/g	B2		-0.51	18821	1.8	18735	18821	18820	148	0.8	15
	J/g	K1		-0.38	28203	1.3	28133	28212	28216	161	0.6	15
Q _{V,gr,d}	J/g	B1		-0.44	22471	1.4	22402	22484	22466	121	0.5	13
	J/g	B2		-0.42	20142	1.4	20083	20167	20136	178	0.9	19
	J/g	K1		-0.35	29137	1	29087	29139	29137	111	0.4	19
S _d	w%	B1		1.67	0.20	15	0.23	0.20	0.20	0.01	7.2	9
	w%	K1		0.65	0.31	15	0.33	0.31	0.31	0.01	3.8	18
V _{db}	w%	B1		1.55	70.2	3	71.8	70.2	70.2	0.8	1.1	7
	w%	B2		1.32	85.2	3	86.9	85.2	85.1	1.3	1.5	11
	w%	K1		-1.55	31.8	4	30.8	31.8	31.8	0.7	2.2	18

Participant 24													
Measurand	Unit	Sample	<div><div></div><div>-303</div><div></div></div>	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
Ash _d	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><di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Participant 25														
Measurand	Unit	Sample	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}		
Ash _d	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-1.73	0.27	30	0.20	0.29	0.28	0.03	9.2	18		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.00	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21		
C _d	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.08	50.2	2.5	50.3	50.2	50.3	0.5	0.9	8		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-0.11	72.3	2.5	72.2	72.2	72.3	0.6	0.9	15		
H _d	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.78	5.93	10	6.16	5.93	5.88	0.24	4.0	9		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.32	4.42	7	4.47	4.39	4.44	0.10	2.4	12		
M _{ad,d}	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>		8.87		8.74	8.87	8.88	0.22	2.4	22		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>		3.47		3.35	3.45	3.47	0.17	5.0	24		
N _d	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>		0.08		<0.07	0.08	0.09	0.04	37.4	7		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.00	2.18	10	2.18	2.18	2.15	0.12	5.5	9		
q _{p,net,d}	J/g	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.00	18821	1.8	18821	18821	18820	148	0.8	15		
	J/g	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-1.20	28203	1.3	27983	28212	28216	161	0.6	15		
q _{V,gr,d}	J/g	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-0.37	20142	1.4	20090	20167	20136	178	0.9	19		
	J/g	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-1.60	29137	1	28904	29139	29137	111	0.4	19		
S _d	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.65	0.31	15	0.33	0.31	0.31	0.01	3.8	18		
V _{db}	w%	B2	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	-0.67	85.2	3	84.4	85.2	85.1	1.3	1.5	11		
	w%	K1	<div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div><div></div></div>	0.00	31.8	4	31.8	31.8	31.8	0.7	2.2	18		

Participant 26													
Measurand	Unit	Sample		z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
Ash _d	w%	K1		0.59	10.8	2.5	10.9	10.8	10.8	0.0	0.4	21	
C _d	w%	K1		-1.00	72.3	2.5	71.4	72.2	72.3	0.6	0.9	15	
EF	t CO2/TJ	K1			94.2		94.0	94.2	94.2	0.2	0.2	5	
M _{ad,d}	w%	K1			3.47		3.63	3.45	3.47	0.17	5.0	24	
q _{p,net,d}	J/g	K1		-0.35	28203	1.3	28139	28212	28216	161	0.6	15	
q _{V,gr,d}	J/g	K1		0.01	29137	1	29139	29139	29137	111	0.4	19	
S _d	w%	K1		0.62	0.31	15	0.32	0.31	0.31	0.01	3.8	18	
V _{db}	w%	K1		1.08	31.8	4	32.5	31.8	31.8	0.7	2.2	18	

Participant 27													
Measurand	Unit	Sample		z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}	
Ash _d	w%	B2		0.49	0.27	30	0.29	0.29	0.28	0.03	9.2	18	
	w%	K1		-2.00	10.8	2.5	10.5	10.8	10.8	0.0	0.4	21	
C _d	w%	B2		-0.32	50.2	2.5	50.0	50.2	50.3	0.5	0.9	8	
	w%	K1		0.90	72.3	2.5	73.1	72.2	72.3	0.6	0.9	15	
EF	t CO2/TJ	K1			94.2		96.5	94.2	94.2	0.2	0.2	5	
H _d	w%	B2		-0.07	5.93	10	5.91	5.93	5.88	0.24	4.0	9	
	w%	K1		-0.23	4.42	7	4.39	4.39	4.44	0.10	2.4	12	
M _{ad,d}	w%	B2			8.87		8.81	8.87	8.88	0.22	2.4	22	
	w%	K1			3.47		3.40	3.45	3.47	0.17	5.0	24	

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Participant 27												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Q _{p.net.d}	J/g	B2		0.58	18821	1.8	18920	18821	18820	148	0.8	15
	J/g	K1		2.17	28203	1.3	28601	28212	28216	161	0.6	15
Q _{V.gr.d}	J/g	B2		0.46	20142	1.4	20208	20167	20136	178	0.9	19
	J/g	K1		2.85	29137	1	29553	29139	29137	111	0.4	19
S _d	w%	K1		-0.43	0.31	15	0.30	0.31	0.31	0.01	3.8	18

Participant 28												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		0.18	5.38	7	5.41	5.45	5.40	0.13	2.4	13
	w%	B2		0.57	0.27	30	0.29	0.29	0.28	0.03	9.2	18
M _{ad.d}	w%	B1			9.10		9.65	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		9.18	8.87	8.88	0.22	2.4	22
Q _{p.net.d}	J/g	B1		0.90	21254	1.5	21398	21254	21264	149	0.7	9
	J/g	B2		0.63	18821	1.8	18929	18821	18820	148	0.8	15
Q _{V.gr.d}	J/g	B1		0.56	22471	1.4	22559	22484	22466	121	0.5	13
	J/g	B2		0.63	20142	1.4	20231	20167	20136	178	0.9	19

Participant 29												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		0.04	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
H _d	w%	K1		0.16	4.42	7	4.45	4.39	4.44	0.10	2.4	12
M _{ad.d}	w%	K1			3.47		3.57	3.45	3.47	0.17	5.0	24
Q _{p.net.d}	J/g	K1		-0.15	28203	1.3	28176	28212	28216	161	0.6	15
Q _{V.gr.d}	J/g	K1		0.01	29137	1	29139	29139	29137	111	0.4	19
S _d	w%	K1		-3.25	0.31	15	0.23	0.31	0.31	0.01	3.8	18
V _{db}	w%	K1		-1.04	31.8	4	31.1	31.8	31.8	0.7	2.2	18

Participant 30												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B2		0.62	0.27	30	0.30	0.29	0.28	0.03	9.2	18
M _{ad.d}	w%	B2			8.87		8.77	8.87	8.88	0.22	2.4	22
Q _{p.net.d}	J/g	B2		0.43	18821	1.8	18895	18821	18820	148	0.8	15
Q _{V.gr.d}	J/g	B2		0.40	20142	1.4	20199	20167	20136	178	0.9	19

Participant 31												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2*s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1		-0.48	5.38	7	5.29	5.45	5.40	0.13	2.4	13
	w%	B2		1.11	0.27	30	0.32	0.29	0.28	0.03	9.2	18
	w%	K1		-0.07	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	B1		-0.80	54.7	2.5	54.2	54.7	54.5	0.5	0.8	6
	w%	B2		0.03	50.2	2.5	50.2	50.2	50.3	0.5	0.9	8
	w%	K1		-0.46	72.3	2.5	71.9	72.2	72.3	0.6	0.9	15
EF	t CO ₂ /TJ	B1			106		106	106	106	1	0.7	4
	t CO ₂ /TJ	K1			94.2		94.4	94.2	94.2	0.2	0.2	5
H _d	w%	B1		0.48	5.74	9	5.86	5.74	5.70	0.21	3.7	6
	w%	B2		0.11	5.93	10	5.96	5.93	5.88	0.24	4.0	9
	w%	K1		-0.44	4.42	7	4.35	4.39	4.44	0.10	2.4	12

Participant 31												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
M _{ad,d}	w%	B1			9.10		8.82	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		8.79	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.54	3.45	3.47	0.17	5.0	24
N _d	w%	B1		1.07	2.71	10	2.86	2.71	2.73	0.08	2.9	6
	w%	B2			0.08		0.07	0.08	0.09	0.04	37.4	7
	w%	K1		0.28	2.18	10	2.21	2.18	2.15	0.12	5.5	9
Q _{p,net,d}	J/g	B1		-0.11	21254	1.5	21237	21254	21264	149	0.7	9
	J/g	B2		-0.29	18821	1.8	18772	18821	18820	148	0.8	15
	J/g	K1		0.05	28203	1.3	28212	28212	28216	161	0.6	15
Q _{V,gr,d}	J/g	B1		0.29	22471	1.4	22517	22484	22466	121	0.5	13
	J/g	B2		-0.44	20142	1.4	20080	20167	20136	178	0.9	19
	J/g	K1		0.13	29137	1	29156	29139	29137	111	0.4	19
S _d	w%	B1		0.72	0.20	15	0.21	0.20	0.20	0.01	7.2	9
	w%	K1		-0.19	0.31	15	0.31	0.31	0.31	0.01	3.8	18
V _{db}	w%	B1		-0.82	70.2	3	69.3	70.2	70.2	0.8	1.1	7
	w%	B2		-0.97	85.2	3	84.0	85.2	85.1	1.3	1.5	11
	w%	K1		-1.08	31.8	4	31.1	31.8	31.8	0.7	2.2	18

Participant 32												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	K1		0.22	10.8	2.5	10.8	10.8	10.8	0.0	0.4	21
C _d	w%	K1		0.24	72.3	2.5	72.5	72.2	72.3	0.6	0.9	15
M _{ad,d}	w%	K1			3.47		3.45	3.45	3.47	0.17	5.0	24
Q _{p,net,d}	J/g	K1		-0.36	28203	1.3	28137	28212	28216	161	0.6	15
Q _{V,gr,d}	J/g	K1		-0.39	29137	1	29081	29139	29137	111	0.4	19
S _d	w%	K1		0.26	0.31	15	0.32	0.31	0.31	0.01	3.8	18
V _{db}	w%	K1		-1.64	31.8	4	30.8	31.8	31.8	0.7	2.2	18

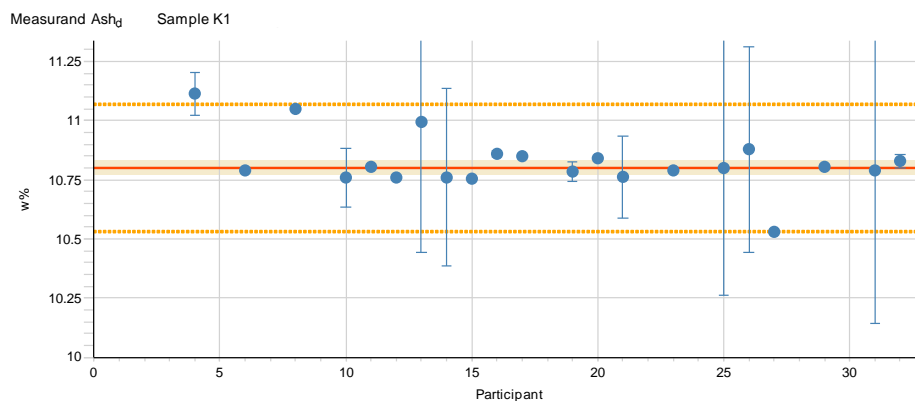
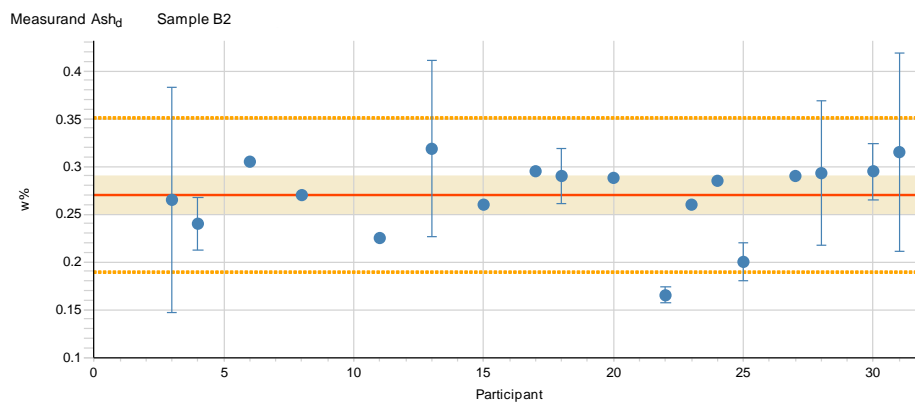
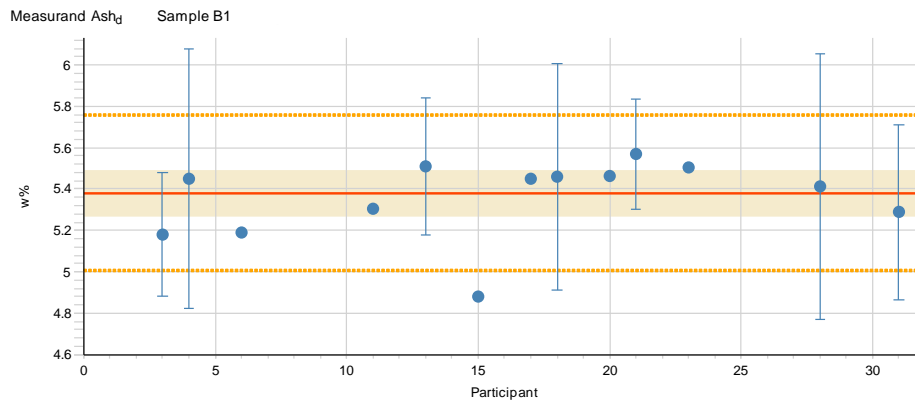
Participant 33												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B1			5.38	7	8.5	5.45	5.40	0.13	2.4	13
	w%	B2			0.27	30	<1	0.29	0.28	0.03	9.2	18
	w%	K1			10.8	2.5	58	10.8	10.8	0.0	0.4	21
M _{ad,d}	w%	B1			9.10		9.70	9.11	9.10	0.34	3.7	15
	w%	B2			8.87		9.15	8.87	8.88	0.22	2.4	22
	w%	K1			3.47		3.45	3.45	3.47	0.17	5.0	24
Q _{V,gr,d}	J/g	B1		-0.22	22471	1.4	22436	22484	22466	121	0.5	13
	J/g	B2		0.18	20142	1.4	20167	20167	20136	178	0.9	19
	J/g	K1		-6.32	29137	1	28216	29139	29137	111	0.4	19

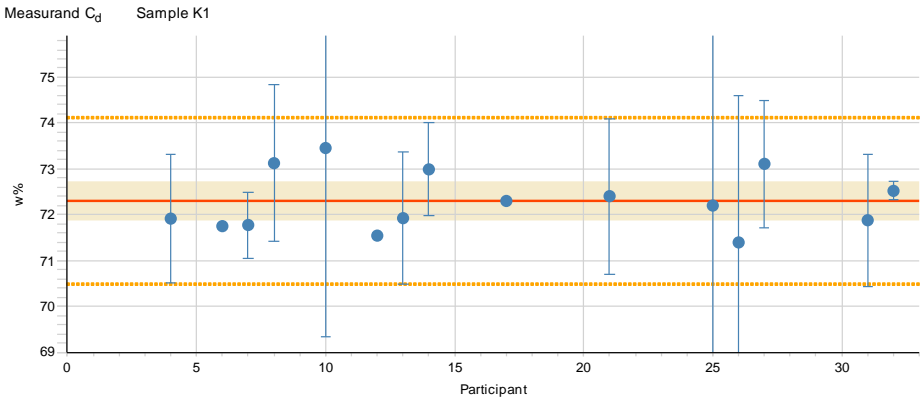
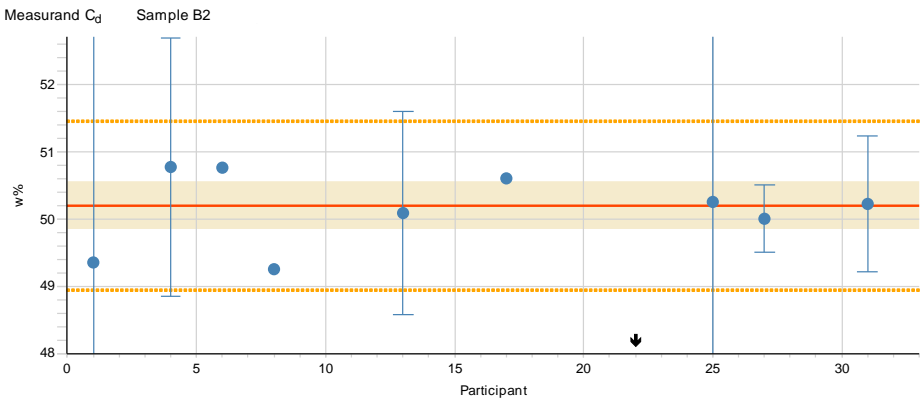
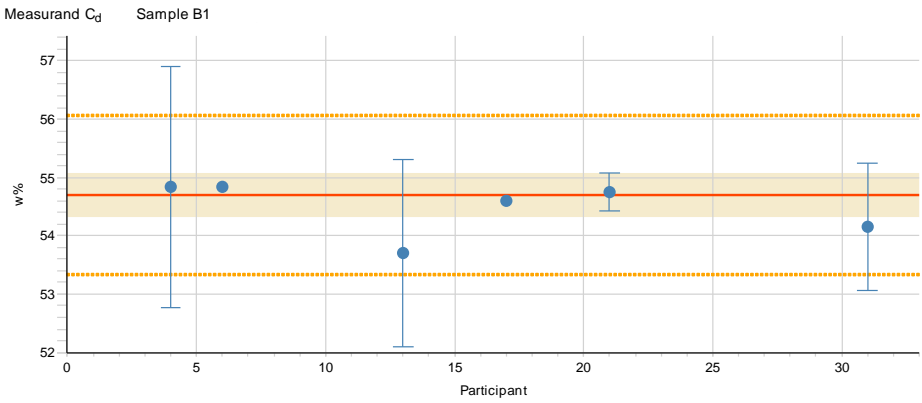
Participant 34												
Measurand	Unit	Sample	-3 0 3	z score	Assigned value	2×s _{pt} %	Participant's result	Md	Mean	s	s %	n _{stat}
Ash _d	w%	B2			0.27	30	<0.1	0.29	0.28	0.03	9.2	18
M _{ad,d}	w%	B2			8.87		9.44	8.87	8.88	0.22	2.4	22
Q _{p,net,d}	J/g	B2		-1.59	18821	1.8	18551	18821	18820	148	0.8	15
Q _{V,gr,d}	J/g	B2		-2.24	20142	1.4	19827	20167	20136	178	0.9	19
V _{db}	w%	B2		-0.16	85.2	3	85.0	85.2	85.1	1.3	1.5	11

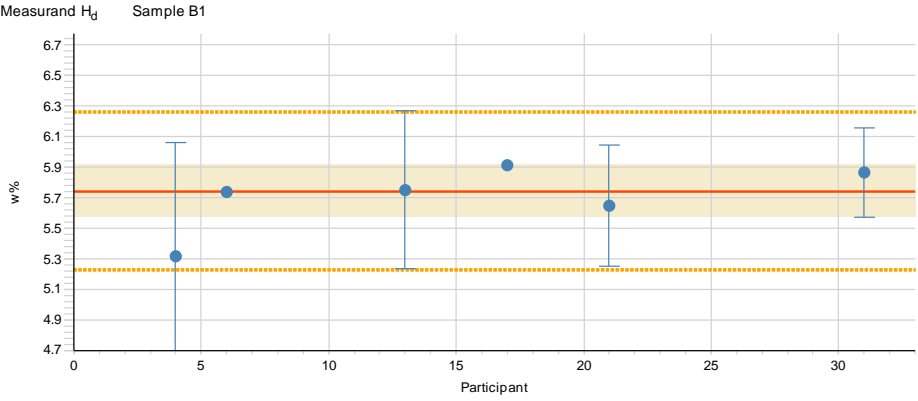
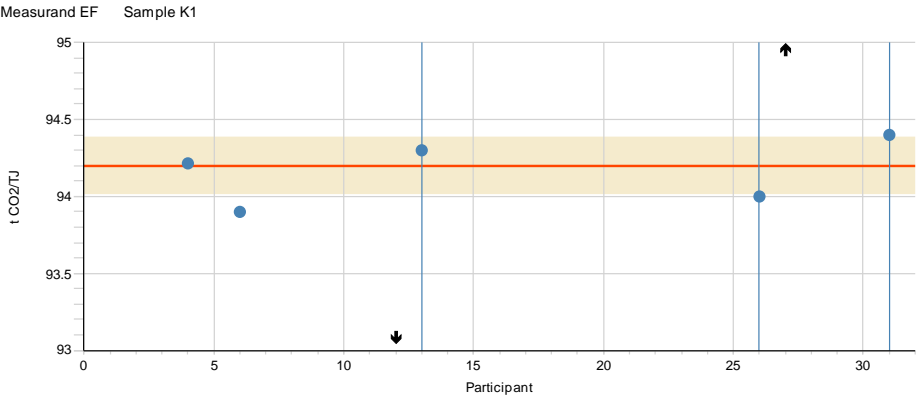
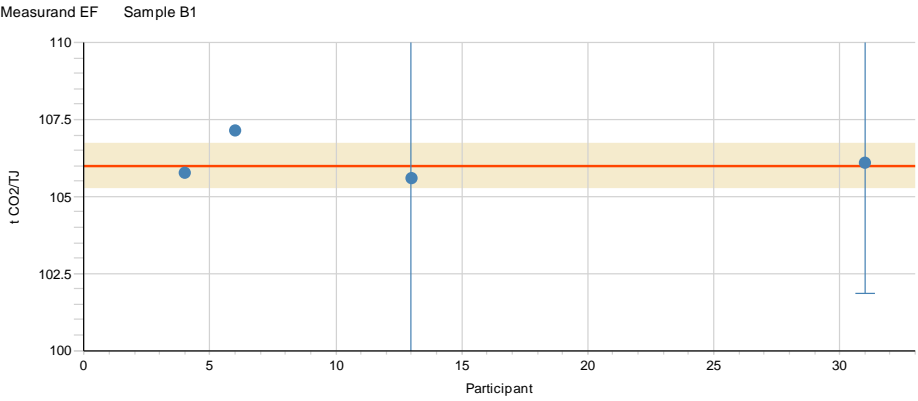
APPENDIX 8: Results of participants and their uncertainties

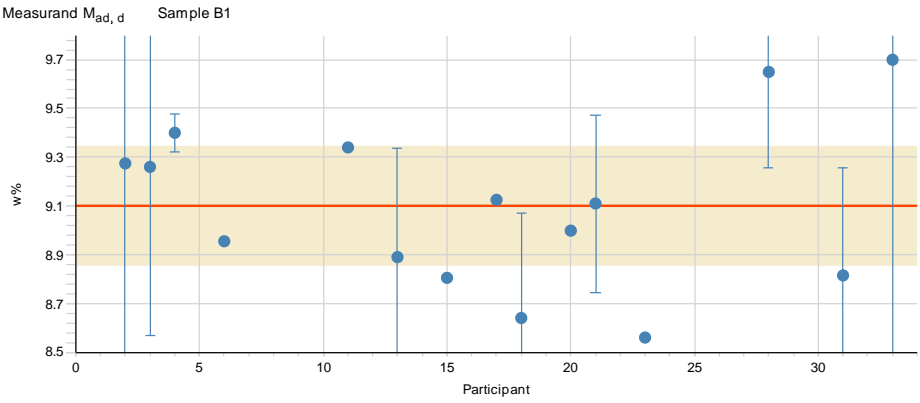
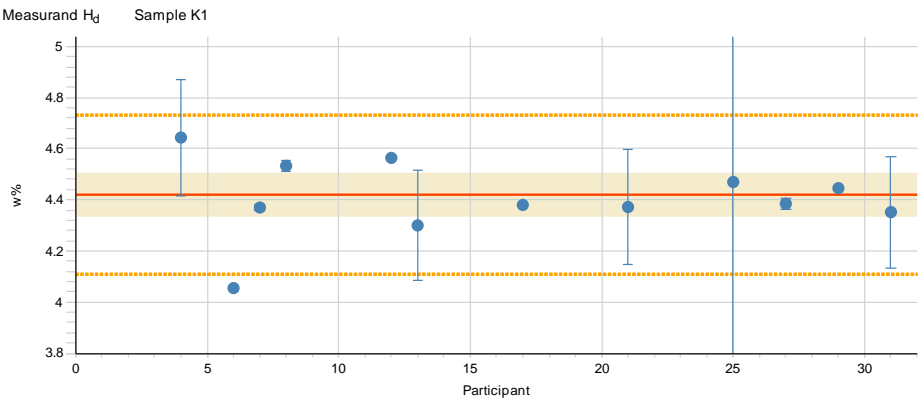
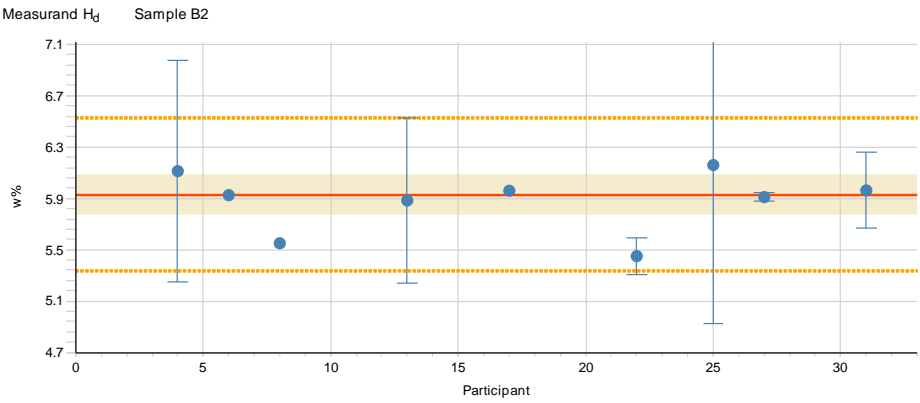
In figures:

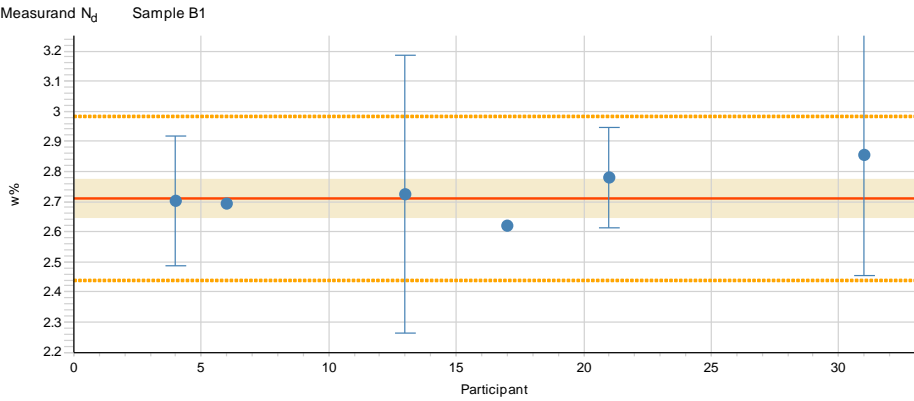
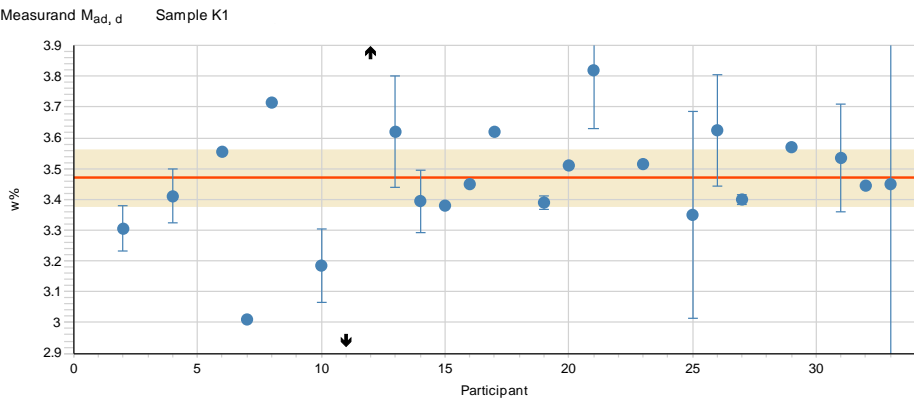
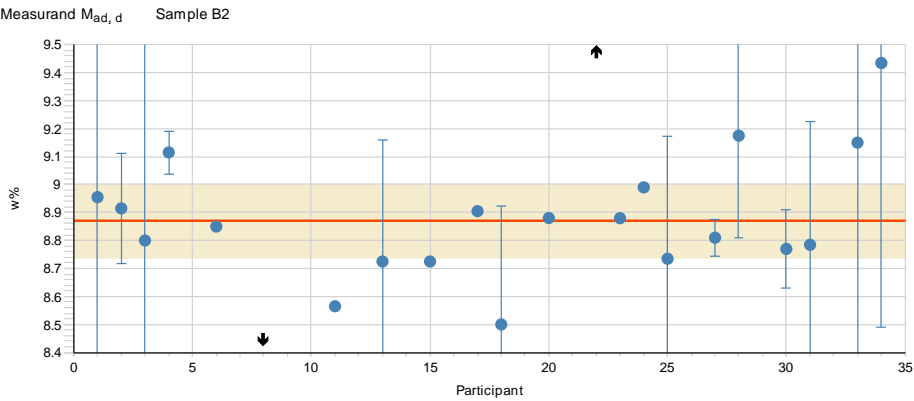
- The dashed lines describe the standard deviation for the proficiency assessment. the red solid line shows the assigned value, the shaded area describes the expanded uncertainty of the assigned value, and the arrow describes the value outside the scale.

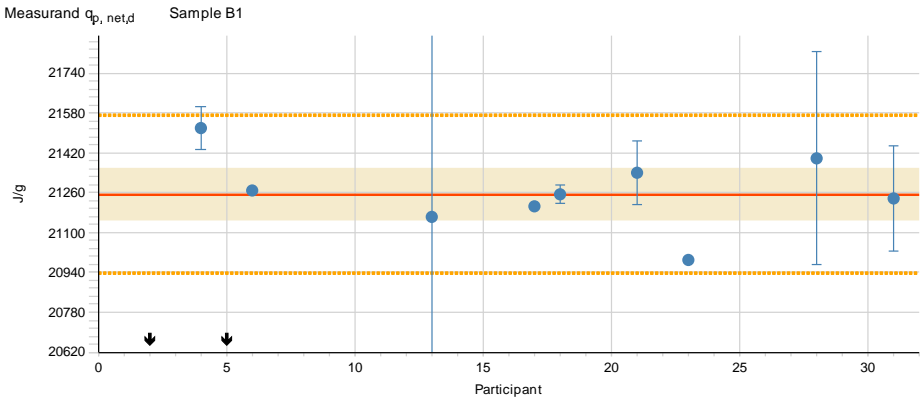
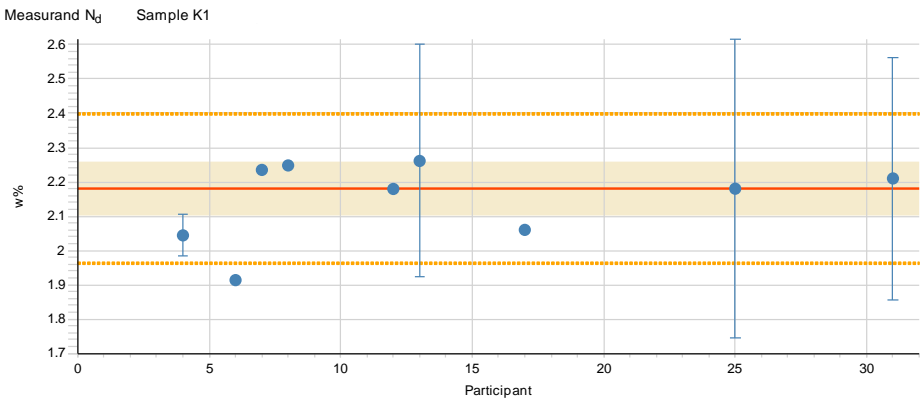
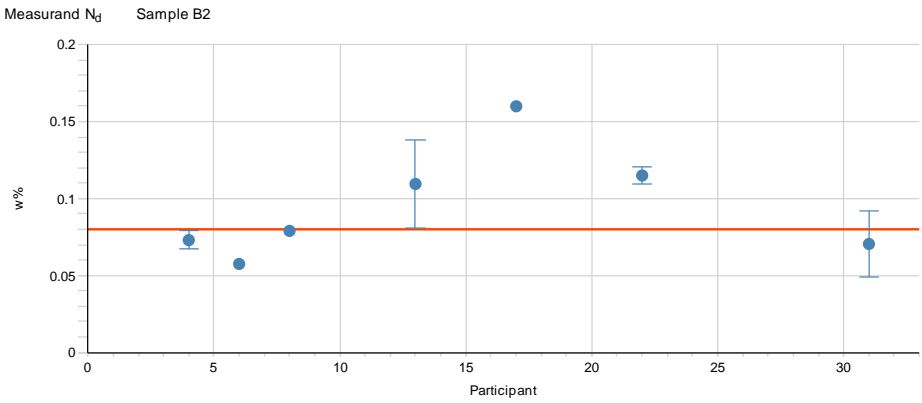


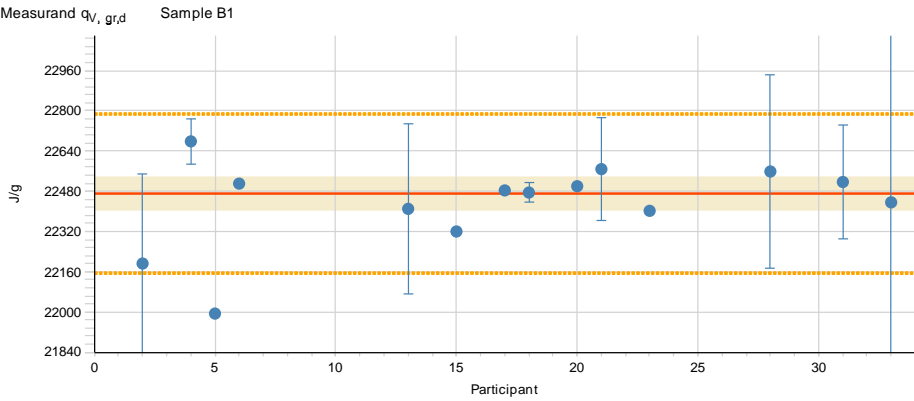
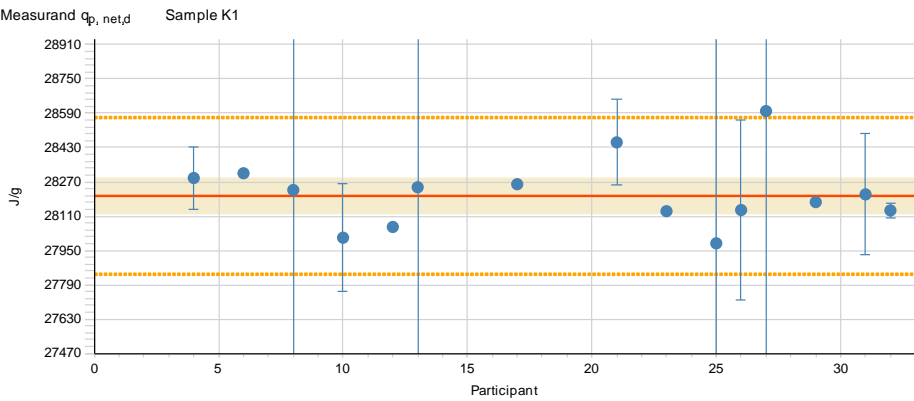
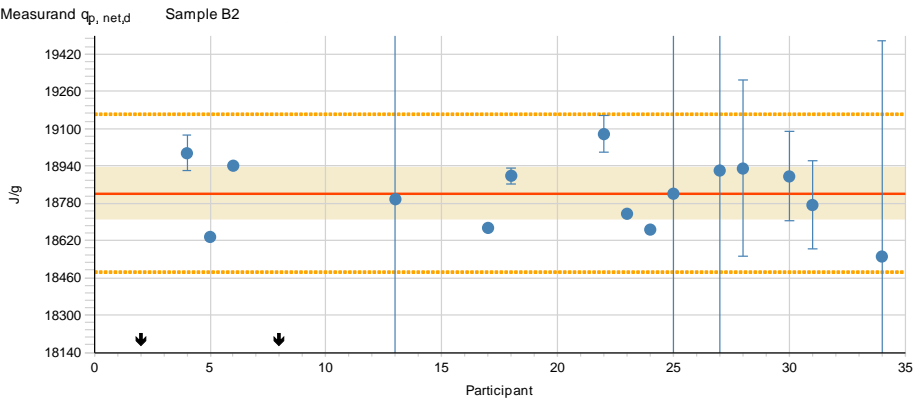


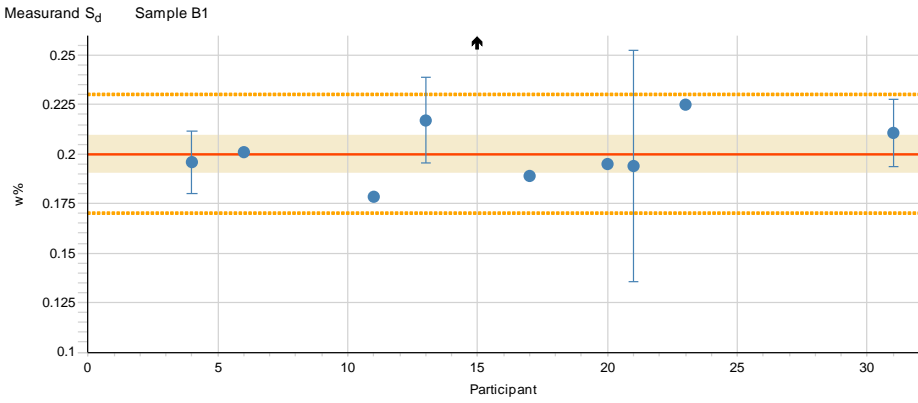
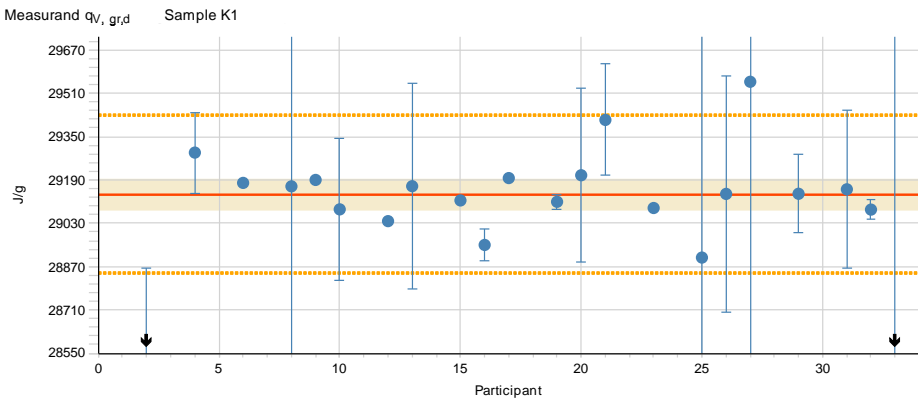
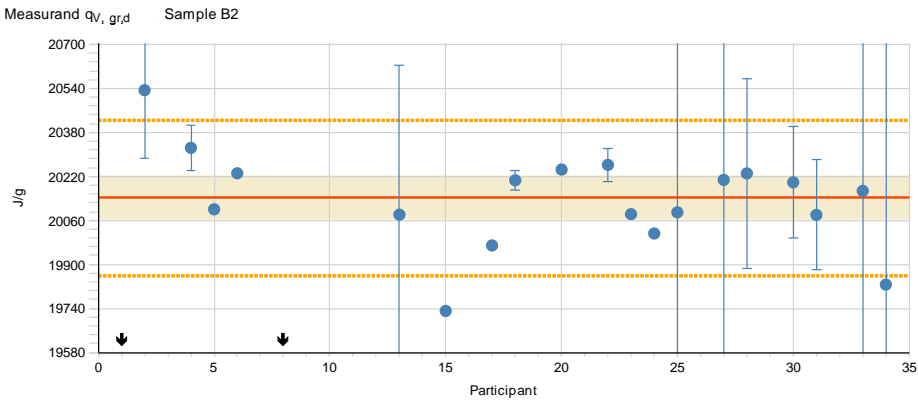


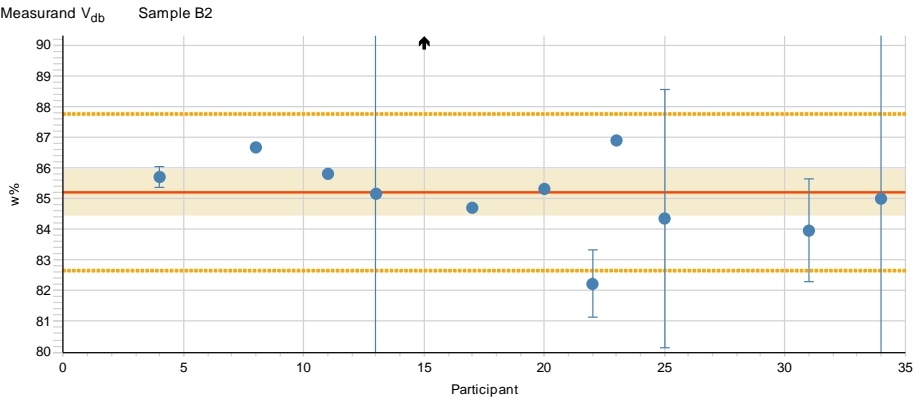
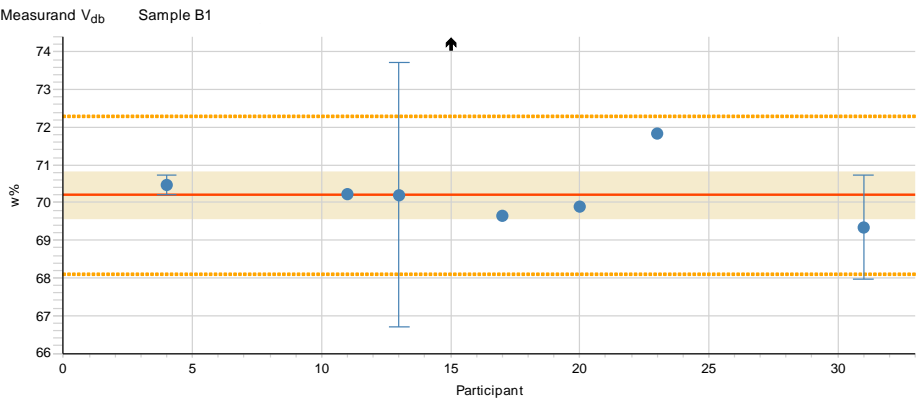
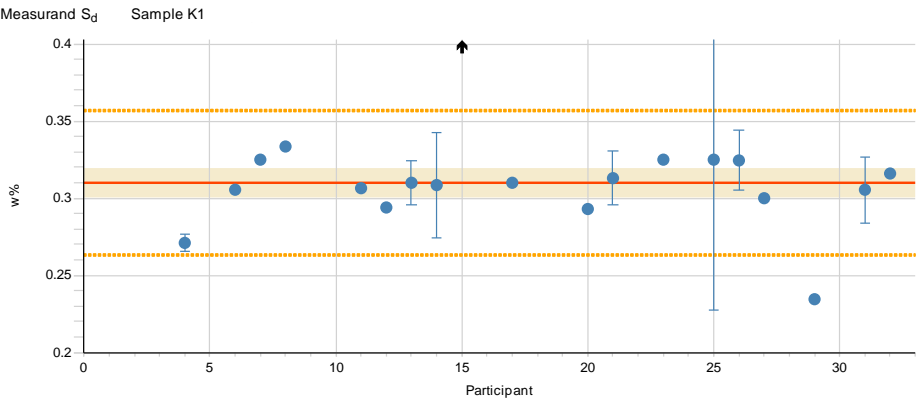


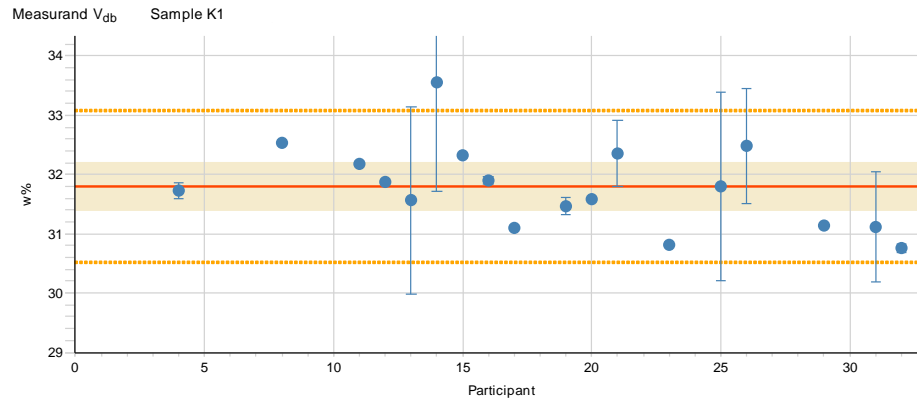












APPENDIX 9: Summary of the z scores

Measurand	Sample	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	%
Ash _d	B1	.	.	S	S	.	S	S	.	S	.	q	.	S	S	.	S	S	.	S	92.3
	B2	.	.	S	S	.	S	.	S	.	.	S	.	S	.	S	.	S	S	.	S	.	q	S	94.4
	K1	.	.	.	Q	.	S	.	S	.	S	S	S	S	S	S	S	S	.	S	S	S	.	S	90.5
C _d	B1	.	.	.	S	.	S	S	.	.	.	S	.	.	.	S	.	.	100
	B2	S	.	.	S	.	S	.	S	S	.	.	.	S	u	.	90.0
	K1	.	.	.	S	.	S	S	S	.	S	.	S	S	S	.	.	S	.	.	.	S	.	.	100
EF	B1
	K1
H _d	B1	.	.	.	S	.	S	S	.	.	.	S	.	.	.	S	.	.	100
	B2	.	.	.	S	.	S	.	S	S	.	.	.	S	S	.	100
	K1	.	.	.	S	.	q	S	S	.	.	.	S	S	.	.	.	S	.	.	.	S	.	.	91.7
M _{ad,d}	B1
	B2
	K1
N _d	B1	.	.	.	S	.	S	S	.	.	.	S	.	.	.	S	.	.	100
	B2
	K1	.	.	.	S	.	q	S	S	.	.	.	S	S	.	.	.	S	88.9
Q _{p,net,d}	B1	.	u	.	S	u	S	S	.	.	.	S	S	.	.	S	.	S	81.8
	B2	.	u	.	S	S	S	.	u	S	.	.	.	S	S	.	.	.	S	S	88.2
	K1	.	.	.	S	.	S	.	S	.	S	.	S	S	.	.	.	S	.	.	.	S	.	S	93.3
Q _{V,gr,d}	B1	.	S	.	S	u	S	S	.	S	.	S	S	.	S	S	.	S	92.9
	B2	u	Q	.	S	S	S	.	u	S	.	q	.	S	S	.	S	.	S	S	76.2
	K1	.	u	.	S	.	S	.	S	S	.	S	S	S	.	S	S	S	.	S	S	S	.	S	86.4
S _d	B1	.	.	.	S	.	S	S	.	S	.	U	.	S	.	.	S	S	.	S	90.0
	K1	.	.	.	S	.	S	S	S	.	.	S	S	S	S	U	.	S	.	.	S	S	.	S	89.5
V _{db}	B1	.	.	.	S	S	.	S	.	U	.	S	.	.	S	.	.	S	87.5
	B2	.	.	.	S	.	.	.	S	.	.	S	.	S	.	U	.	S	.	.	S	.	q	S	83.3
	K1	.	.	.	S	.	.	.	S	.	.	S	S	S	Q	S	S	S	.	S	S	S	.	S	94.4
% accredited		50	20	100	95	50	89	100	86	100	100	100	100	100	75	45	100	100	100	100	100	100	50	100	
		1	4	2	22	4	19	1	3	1	3			22	1		3	22		3	1	12		2	

Measurand	Sample	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45	46	%
Ash _d	B1	S	.	.	S	92.3
	B2	<i>S</i>	S	.	<i>S</i>	S	.	<i>S</i>	S	94.4
	K1	.	S	S	<i>q</i>	.	<i>S</i>	.	S	<i>S</i>	90.5
C _d	B1	S	100
	B2	.	S	.	S	.	.	.	S	90.0
	K1	.	S	S	S	.	.	.	S	<i>S</i>	100
EF	B1	
	K1	
H _d	B1	S	100
	B2	.	S	.	S	.	.	.	S	100
	K1	.	<i>S</i>	.	S	.	<i>S</i>	.	S	91.7
M _{ad,d}	B1	
	B2	
	K1	
N _d	B1	S	100
	B2	
	K1	.	S	S	88.9
Q _{p,net,d}	B1	S	.	.	S	81.8
	B2	<i>S</i>	S	.	S	S	.	<i>S</i>	S	.	.	<i>S</i>	88.2
	K1	.	S	S	Q	.	<i>S</i>	.	S	<i>S</i>	93.3
Q _{V,gr,d}	B1	S	.	.	S	.	S	92.9
	B2	<i>S</i>	S	.	S	S	.	<i>S</i>	S	.	S	<i>q</i>	76.2
	K1	.	S	S	Q	.	S	.	S	<i>S</i>	<i>u</i>	86.4
S _d	B1	S	90.0
	K1	.	S	S	S	.	<i>u</i>	.	S	<i>S</i>	89.5
V _{db}	B1	S	87.5
	B2	.	S	S	.	.	<i>S</i>	83.3
	K1	.	<i>S</i>	S	.	.	<i>S</i>	.	S	<i>S</i>	94.4
% accredited		100	100	100	73	100	83	100	100	100	67	67													
			12	6	9	6	1		22		3	1													

S - satisfactory ($-2 \leq z \leq 2$), Q - questionable ($2 < z < 3$), q - questionable ($-3 < z < -2$),

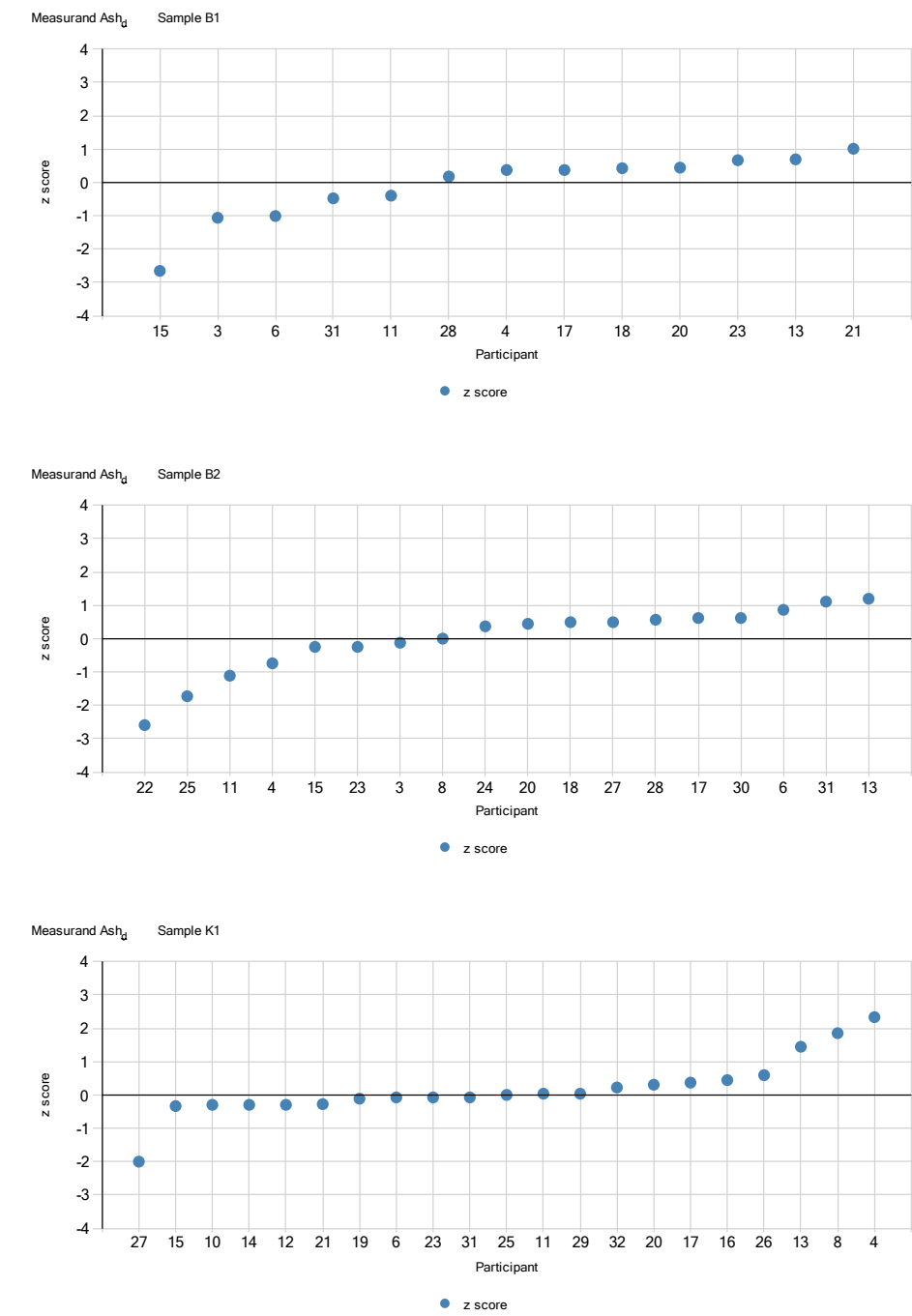
U - unsatisfactory ($z \geq 3$), and u - unsatisfactory ($z \leq -3$), respectively

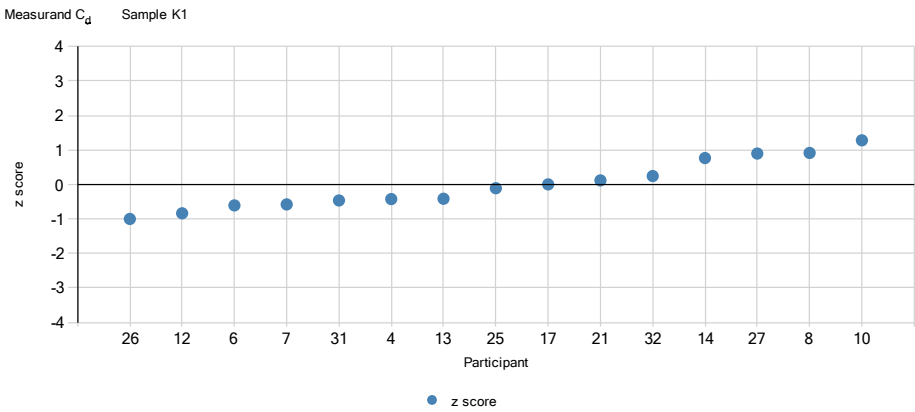
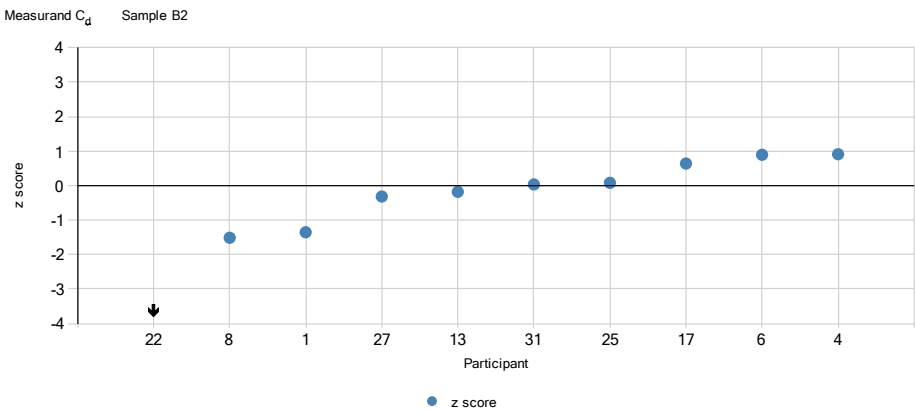
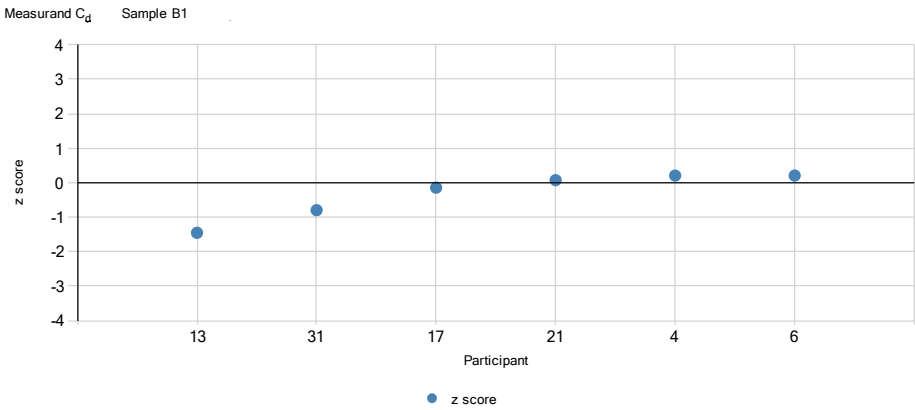
bold - accredited, italics - non-accredited, normal - unknown

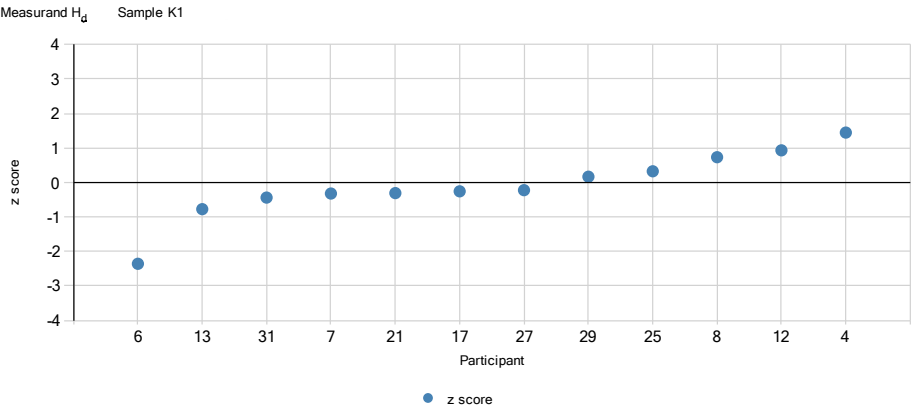
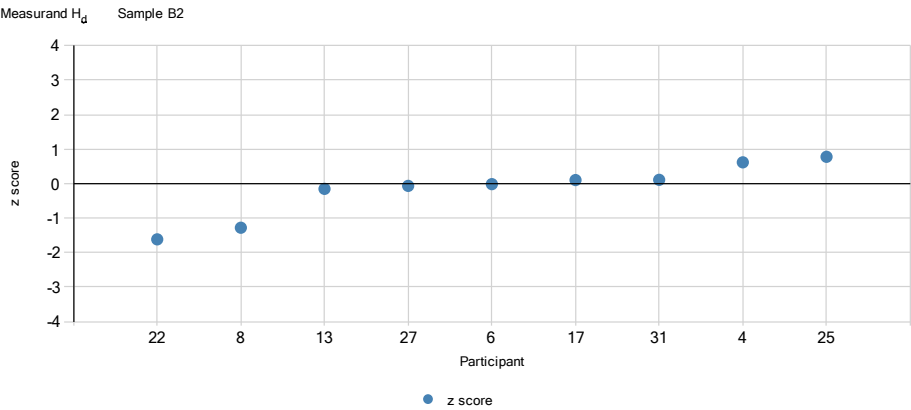
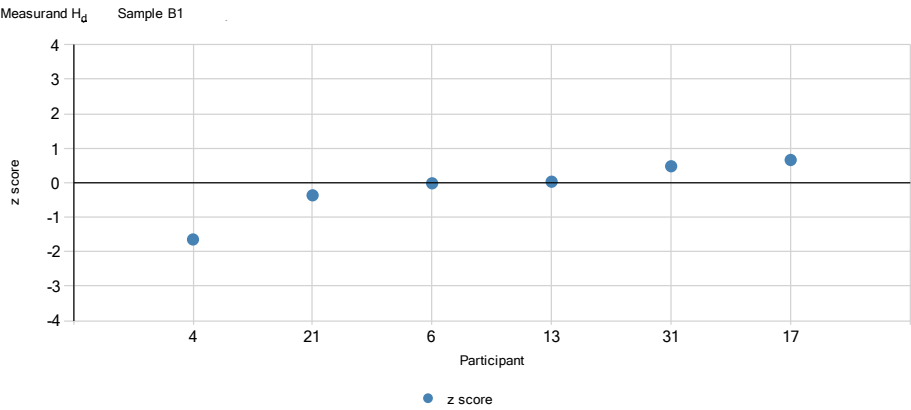
% - percentage of satisfactory results

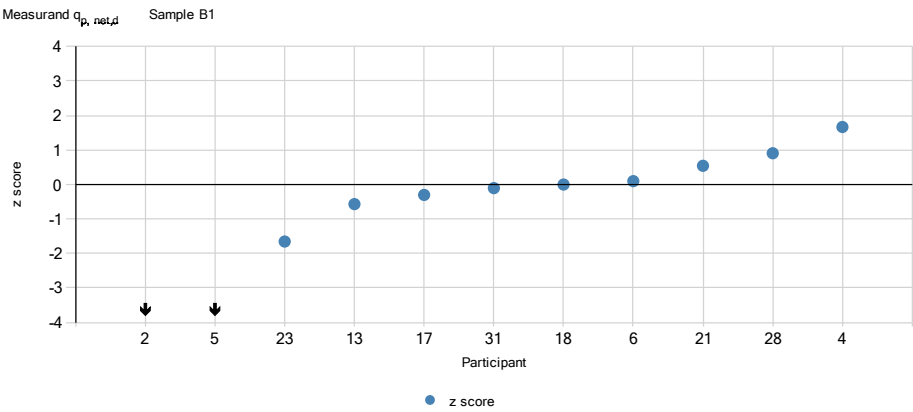
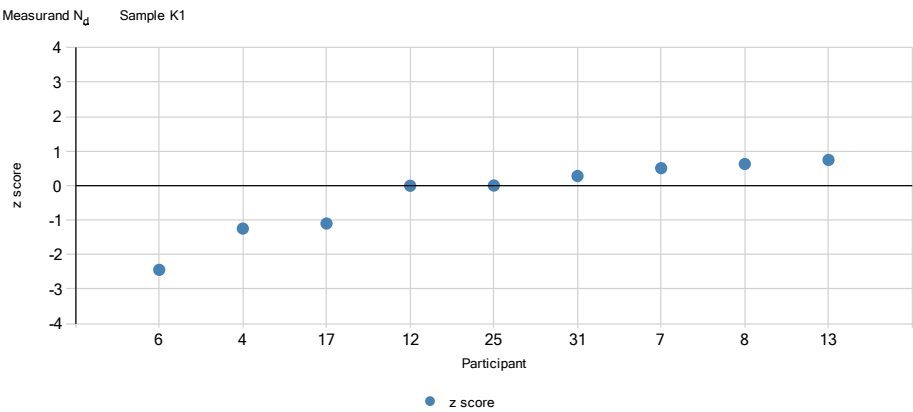
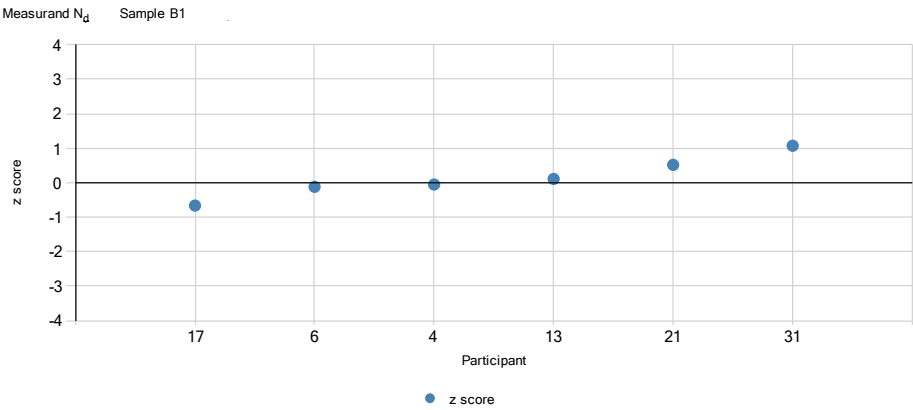
Totally satisfactory, % in all: 90 % in accredited: 94 % in non-accredited: 84

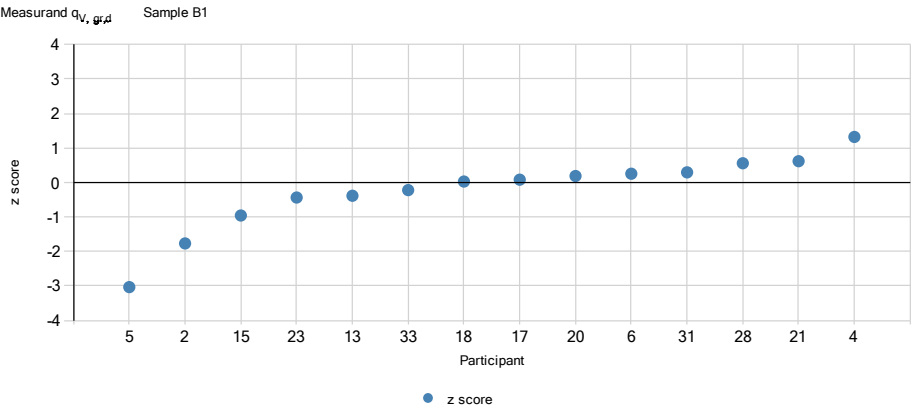
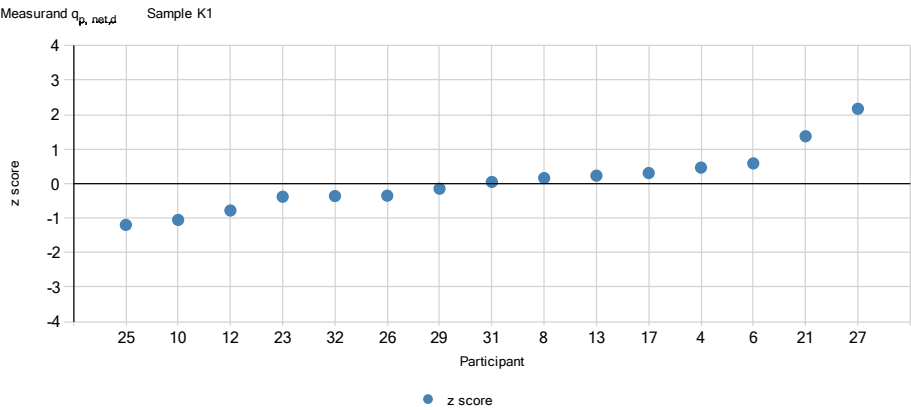
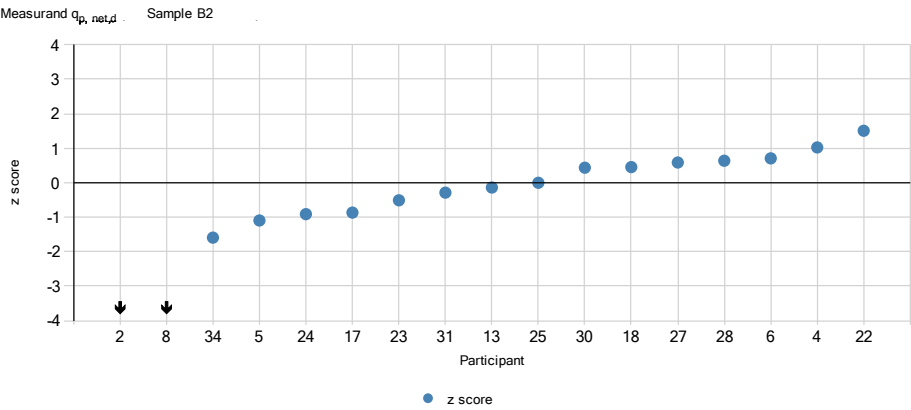
APPENDIX 10: z scores in ascending order

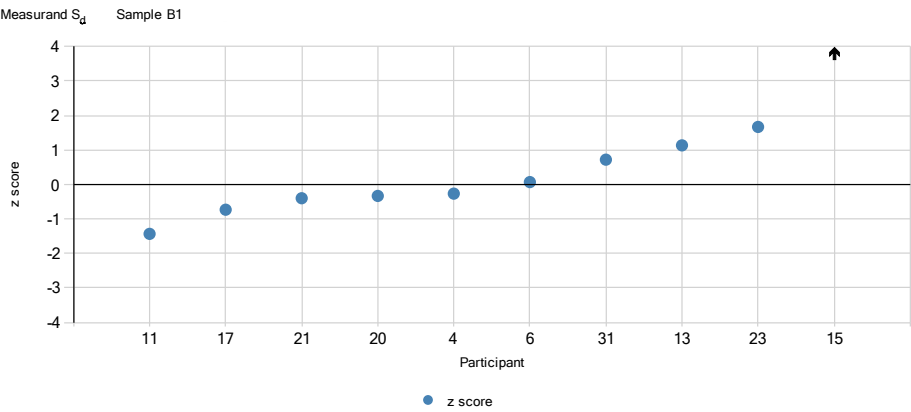
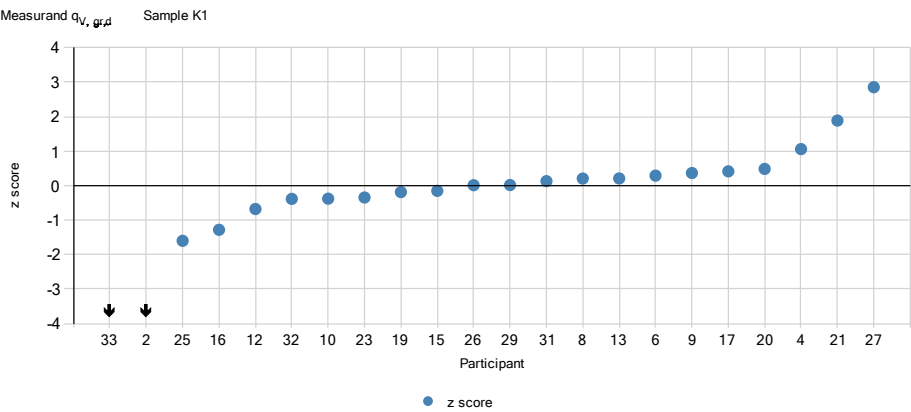
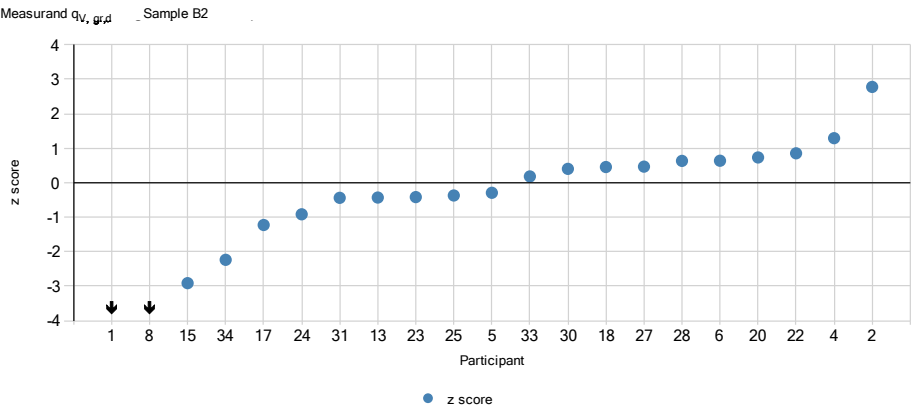


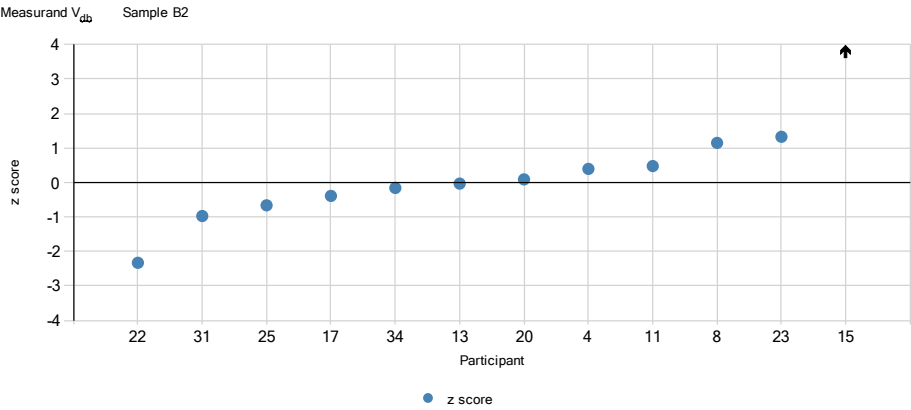
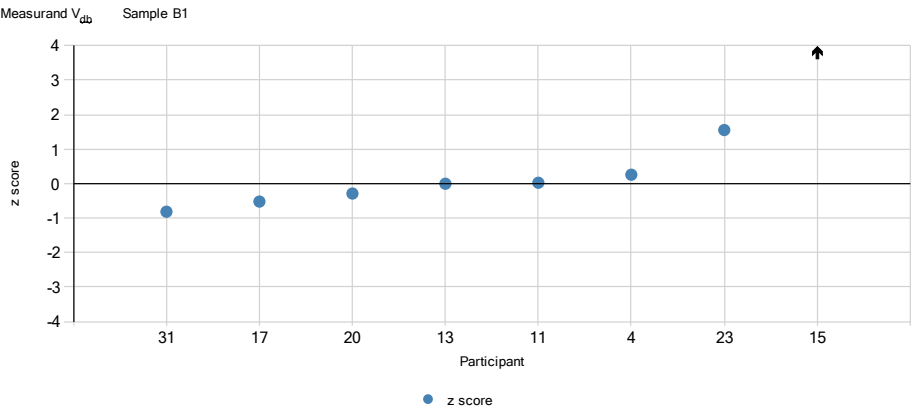
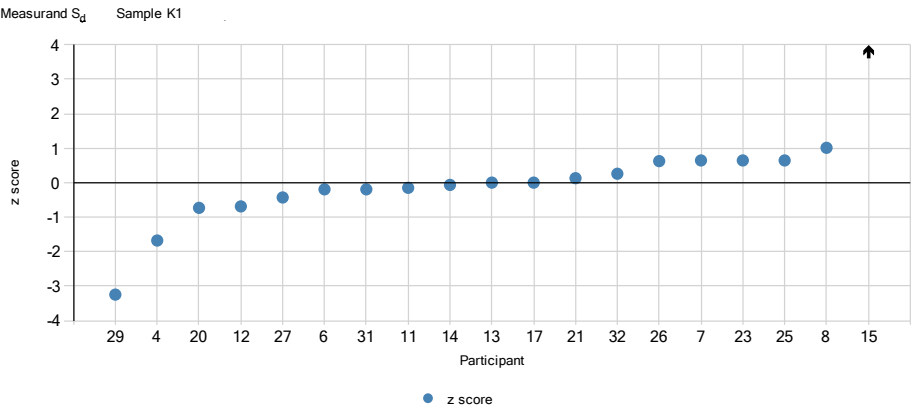


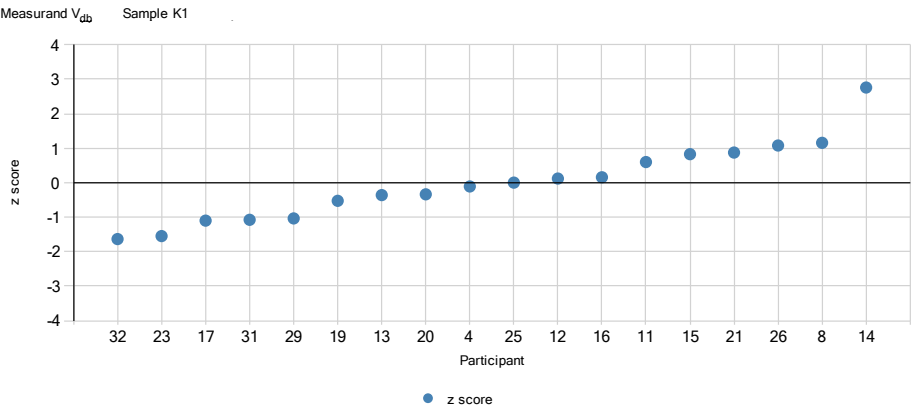












APPENDIX 11: Analytical measurements and background information for calculations

Reported details of the measurements:

Analysis carried out from:	Sample B1 (peat)	Sample B2 (wood pellet)	Sample K1 (coal)
Air dried samples:	participants 13, 15, 18, 20, 21	participants 13, 15, 18, 20, 22, 24, 27, 30	participants 9, 10, 13, 14, 15, 16, 20, 21, 26, 27, 29, 31
Drying in 105 °C:	participants 28, 31	participants 3, 8, 28, 31	participants 7, 8, 12, 14, 19, 29, 32
Other:	participants 3: 108°C degrees 4: original samples 6: as received 11: not dried sample	participants 4: original samples 6: as received 11: not dried sample	participants 4: original samples 6: as received 11: not dried sample

Correction taken into account in calculations:

Gross calorific value			
Participants and correction factors used	Sample		
	B1 (peat)	B2 (wood pellet)	K1 (coal)
4: wire	x	x	x
6: wire, ignition, S, analysis moisture	x	x	x
8: wire, ignition, S, acid correction		x	x
9: wire, ignition, analysis moisture			x
10: wire, ignition, analysis moisture			x
11: wire, S, N, analysis moisture	x	x	x
12: S, N			x
13: wire, ignition, acid correction, analysis moisture	x	x	x
13: S	x		x
15: wire, analysis moisture	x	x	x
15: S	x		x
15: N			x
16: analysis moisture			x
18: wire, ignition, analysis moisture	x	x	
19: wire, ignition			x
20: wire, ignition, S, analysis moisture	x	x	x
21: wire, ignition, S, analysis moisture	x		x
21: N	x		
21: acid correction			x
22: wire, ignition, S, N, analysis moisture		x	
24: wire, ignition, S, N, analysis moisture		x	
26: wire, S, analysis moisture			x
27: wire, S, acid correction		x	x
28: wire, ignition, acid correction	x	x	
29: wire, S, acid correction, analysis moisture			x
30: ignition, S, N, acid correction. analysis moisture		x	
31: wire, S, acid correction	x	x	x
31: analysis moisture			x
32: ignition, acid correction, analysis moisture			x

Correction taken into account in calculations:

Net calorific value (literature value in brackets)			
Participant	Sample		
	B1 (peat)	B2 (wood pellet)	K1 (coal)
6	N+O, H	N+O, H	N+O, H
8		H	H
10			H literature 4.75
12			O. H
13	N+O, H	N+O, H	N+O, H
18	H calculated	H calculated	
21	N+O, H		H
22		N+O literature (42.88+0.0997)	
26			H calculated
27		N+O, H	H
28	N+O literature (35/40), H (5.6/6.0)	N+O literature (35/40), H (5.6/6.0)	
29			N+O, H
30		literature N+O 42+0.1 and literature H 6.0	
31	N+O, H		literature values of N+O
32			ISO 17247

Methods used in ash_d and moisture (M_{ad}) measurements:

Ash _d			Sample B1 (peat)	Sample B2 (wood pellet)	Sample K1 (coal)
Sample amount (g)			participants 3: 1 4: 1 6: 1.5 11: 1 13: 1.9 15: 0.8 18: 1 20: 1 28: 2 31: 1	participants 3: 1 4: 1 6: 1.5 8: 1 11: 1 13: 1.9 15: 0.8 18: 1 20: 1 22: 2 24: 1.00 27: 1 28: 2 30: 1 31: 1	participants 4: 1 6: 1.5 8: 10 10: 1.00 11: 1 12: 1 13: 1.9 14: 1 15: 1 16: 1 19: 0.97 20: 1 26: 1 27: 1 29: 1 31: 1 32: 1
Measurement	Method	°C	Sample B1 (peat)	Sample B2 (wood pellet)	Sample K1 (coal)
Ash content (ashing temperature °C)	Gravimetric:	500		part 27	
		550	parts 4, 11, 13, 18, 20, 21, 28	parts 3, 4, 8, 11, 13, 18, 20, 22, 24, 28, 30	
		700			part 8
		710			part 16
		815	part 3		parts 4, 10, 11, 12, 13, 19, 20, 2, 29, 31
	TGA:	550	parts 6, 15, 31	parts 6, 15, 22, 31	part 6
		750			parts 14, 21, 26
		815			parts 15, 31, 32
	Other:			part 24: ISO 18122: 2016	
Moisture content of analysis sample, M _{ad} (temperature °C)	Air:		parts 3, 4, 11, 13, 15, 18, 20, 21, 28, 31	parts 3, 4, 8, 11, 13, 15, 18, 20, 22, 24, 27, 28, 30, 31	parts 4, 10, 11, 12, 14, 15, 16, 19, 20, 21, 27, 29
	N ₂ atmosphere:		part 6	parts 6, 22	parts 6, 7, 8, 13, 21, 26, 31, 32
	Gravimetric:	105	parts 3, 4, 11, 18, 20, 21, 28	parts 3, 4, 11, 13, 18, 20, 22, 24, 27, 28, 30	parts 4, 10, 11, 12, 13, 14, 16, 19, 20, 21, 27, 29
		107		part 8	part 8
		108	part 3		
	TGA:	105	parts 6, 15, 31	parts 6, 15, 22, 31	parts 6, 15, 21, 31, 32
		107			part 26
	Other			part 24: ISO 18134-3:2016	

CHN-measurements carried out by:

Sample			
	B1	B2	K1
Air dried samples:	parts 13, 31	parts 13, 22, 27,31	parts 10,13, 21, 26, 27, 29, 31
Drying in 105 °C:	part 21	parts 8	parts 7, 8, 12, 14, 29, 32
Other	part 6: As received and correction for moisture	part 6: As received and correction for moisture	part 6: As received and correction for moisture

Detection limits in nitrogen and sulphur measurements:

Participant	Detection limit for N (w%)	Participant	Detection limit for S (w%)
4	0.10	4	0.01
6	0.02	6	0.02
12	0.001	12	0.001
13	0.1	13	0.03
22	50*	14	0.05
29	0.015	15	0.1
		20	0.01
		21	0.06
		22	15000*
		26	0.01
		27	0.01
		29	0.2
		31	0.01
		32	7.0*

*Possible high values are reported invalid values. Detection limits reported by participants 22 and 32 conflict with reported results.

Calculations of Emission factor (EF)¹:**We have used the equation based on the decision EU601/2012(21.6.2012).****If no, describe how?**

	Sample B1 (peat)	Sample K1 (coal)
Yes:	parts 4, 31	parts 4, 12, 26, 27, 31
No:	parts 6 (national guide), 13, 18	parts 6 (national guide), 13, 18, 32

¹In the cover letter the provider gave the participants the possibility to calculate the EF-value using the procedure presented in the EC directive and using the total moisture content as presented in the letter. Later it was obtained, that the EC directive is not giving the detailed equation for calculation of EF-values. Therefore, some national guides for the equation of EF value calculation have been produced. As a result from this, the Energy Authority in Finland has made the guideline for the calculation of emission factor for fossile fuels as follows:

$$EF = 1000 \times 3.664 \times (C/100) \times (1 - M_{ar}/100)/Q_{net.ar}, \text{ where}$$

EF emission factor, g CO₂/MJ

C carbon content as dry, %

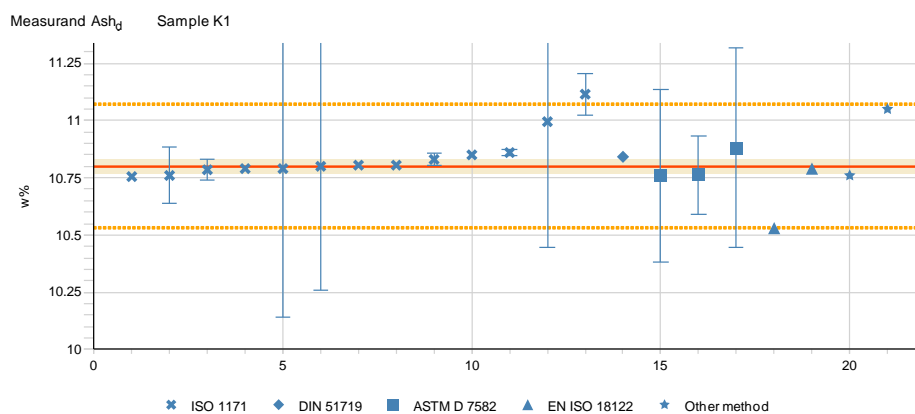
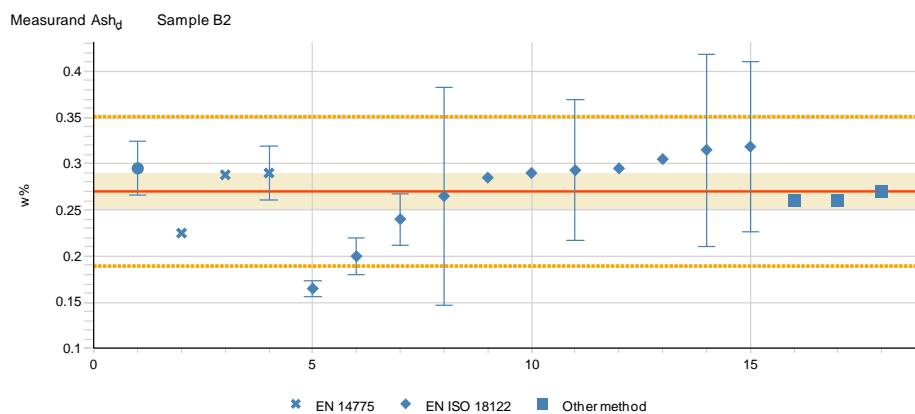
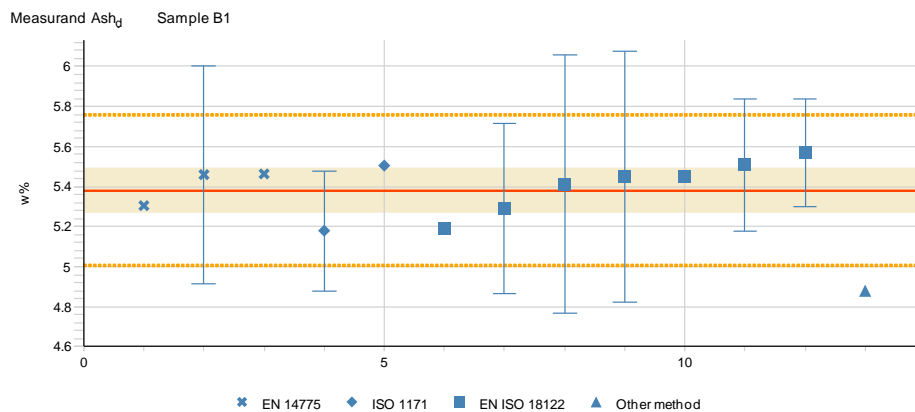
M_{ar} total moisture as received, %Q_{net.ar} net calorific value as received, MJ/kg

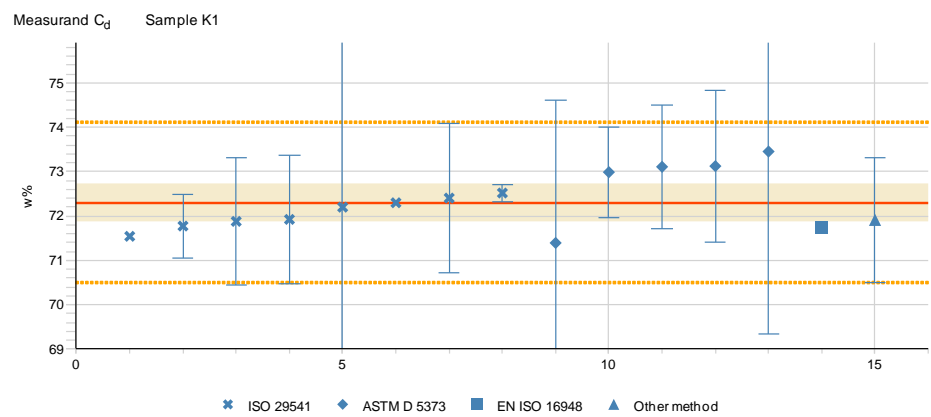
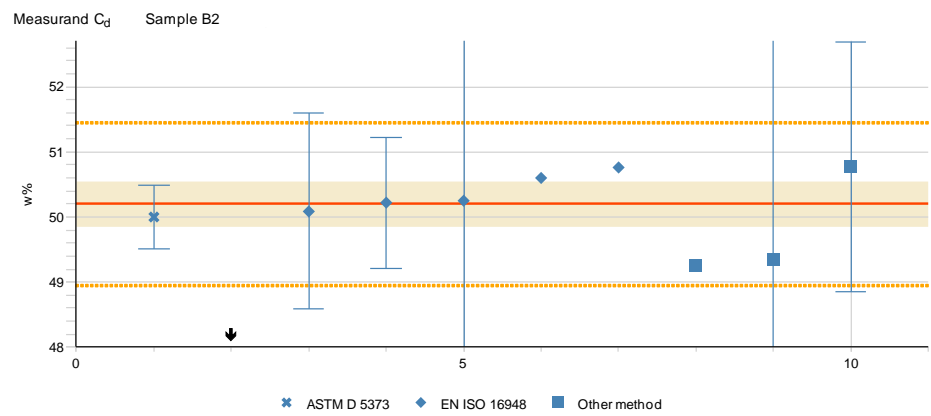
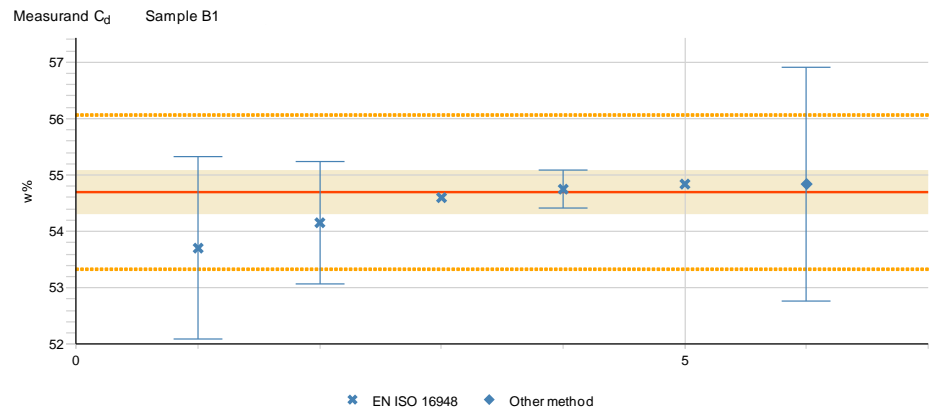
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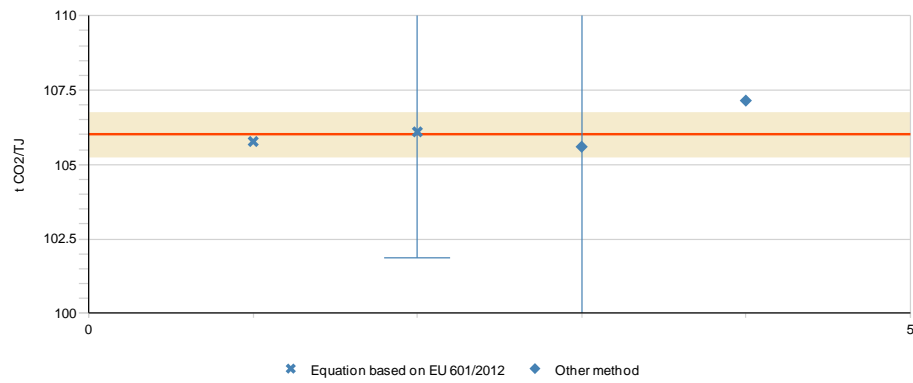
APPENDIX 12: Results grouped according to the methods

The explanations for the figures are described in the Appendix 8. The results are shown in ascending order.

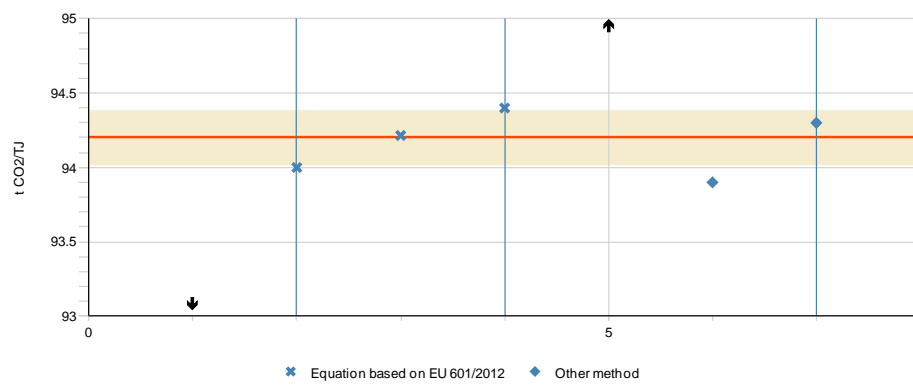
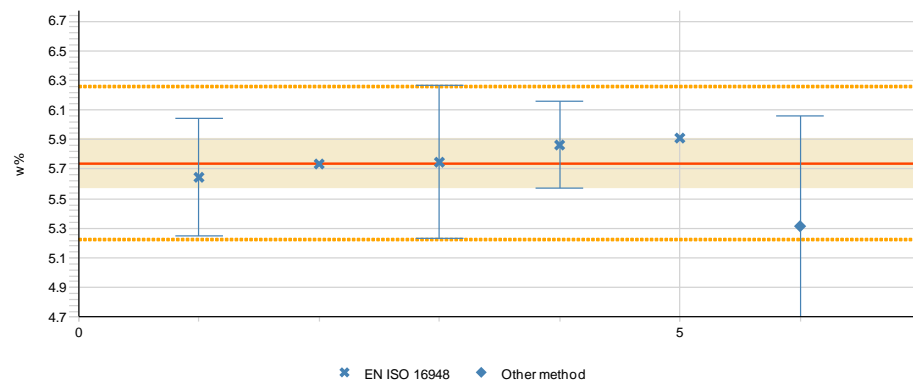


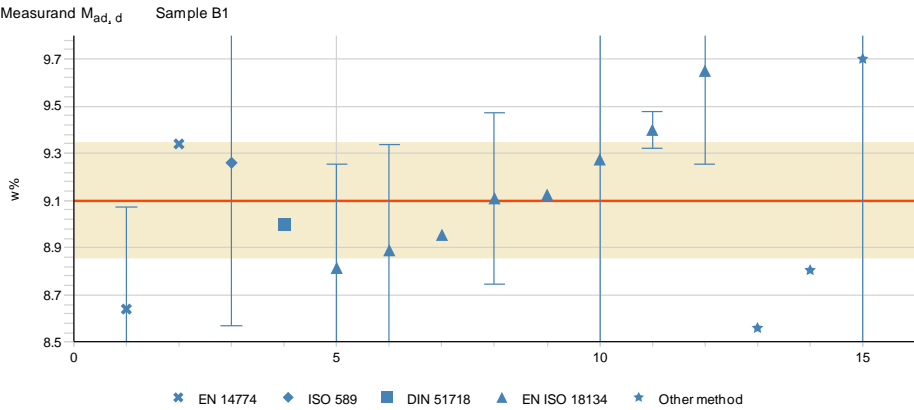
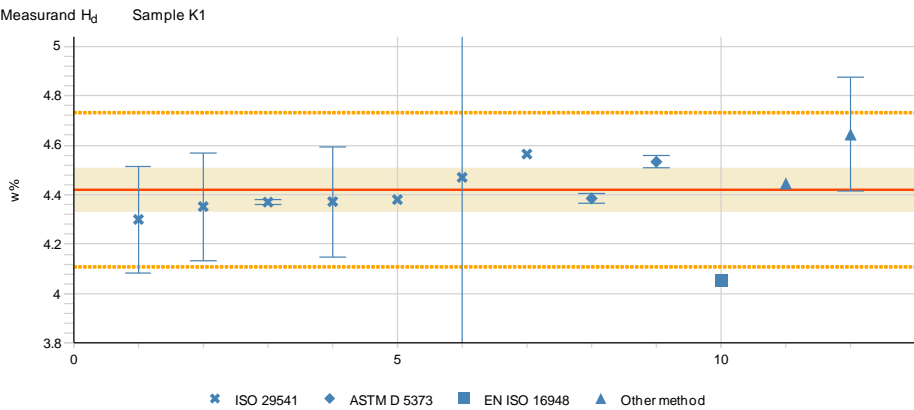
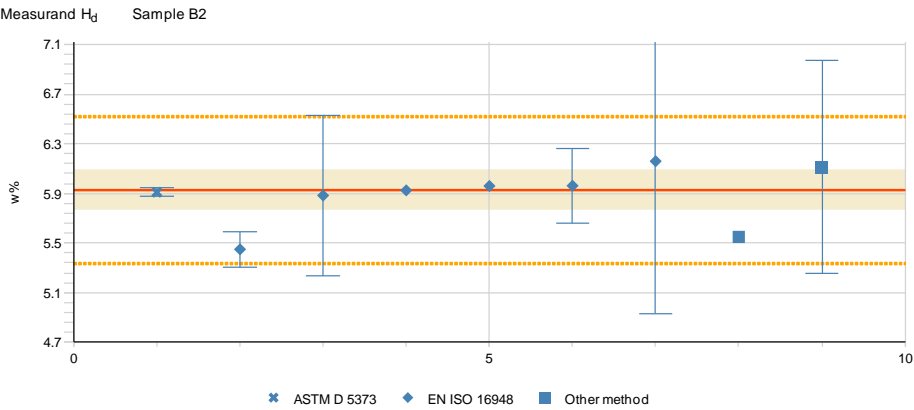


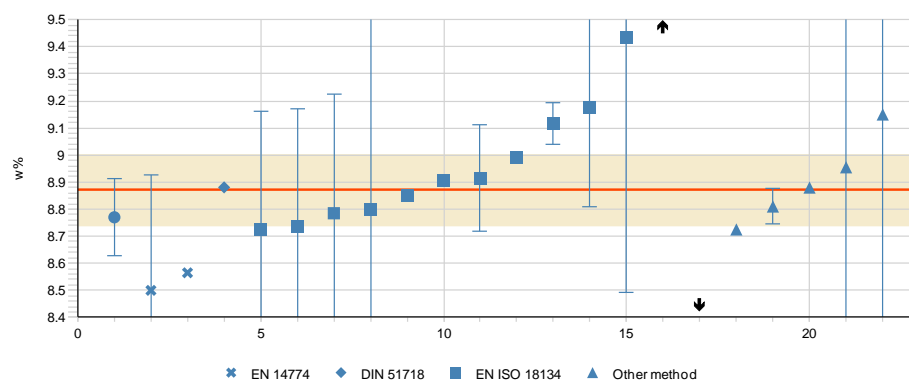
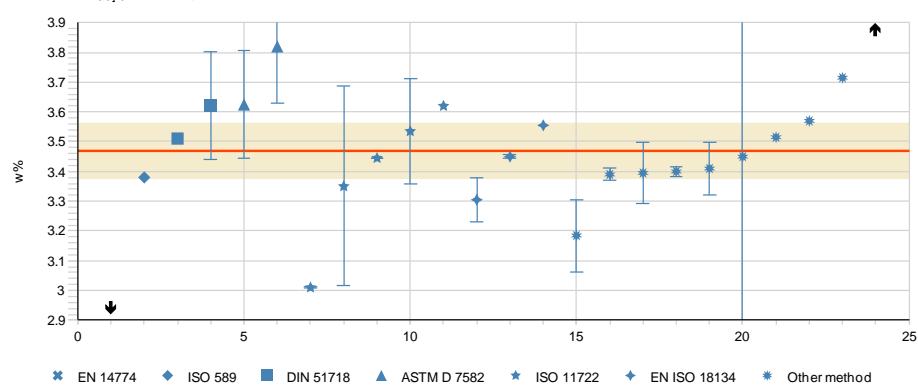
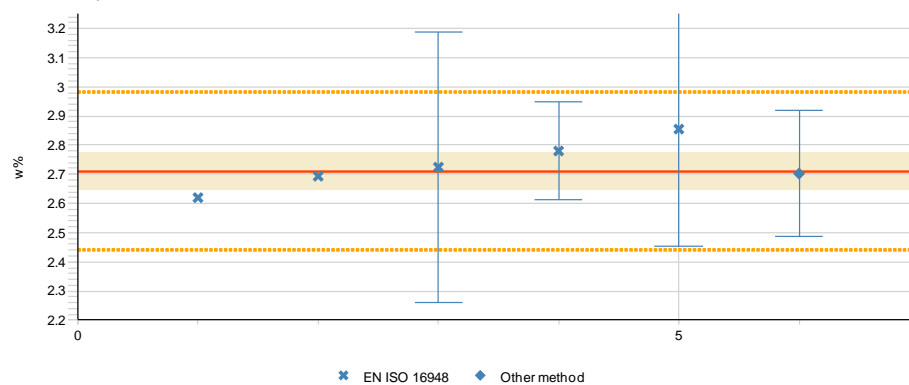
Measurand EF Sample B1

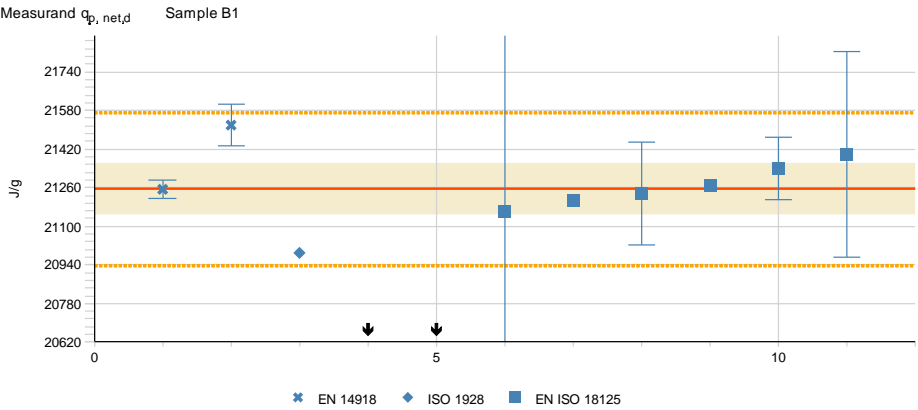
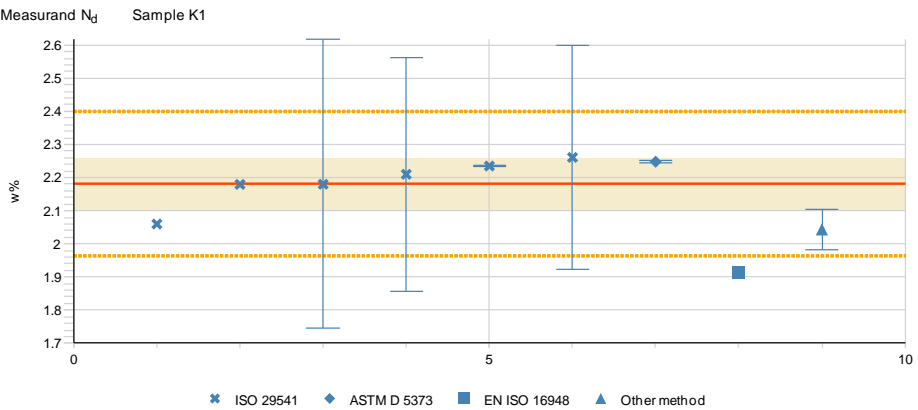
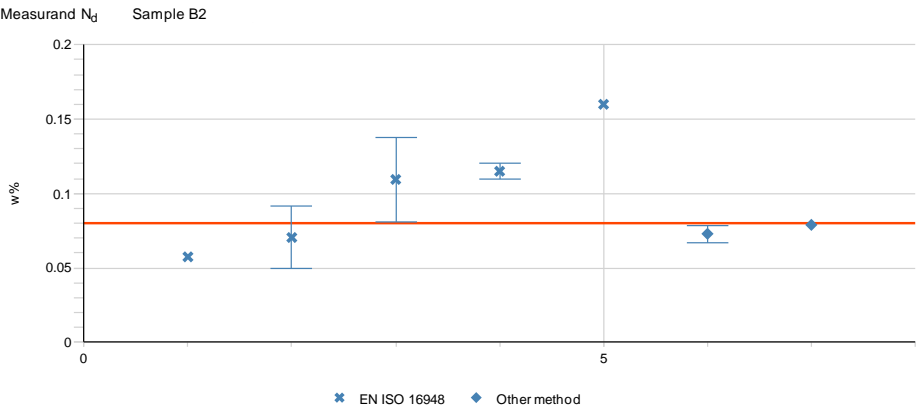


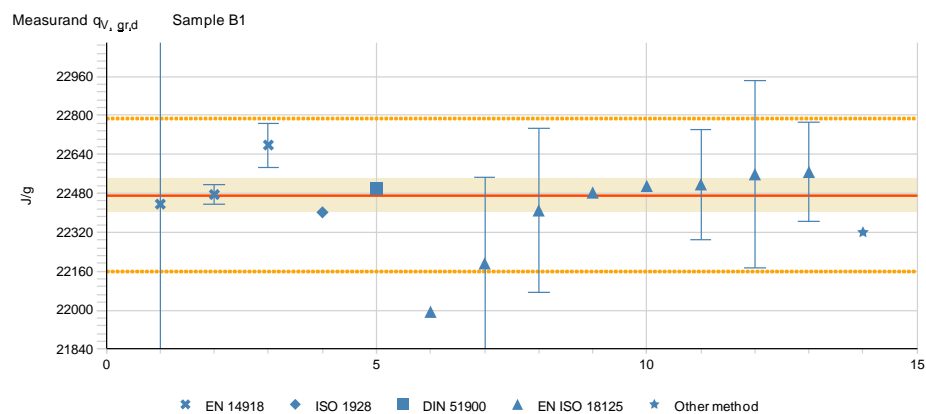
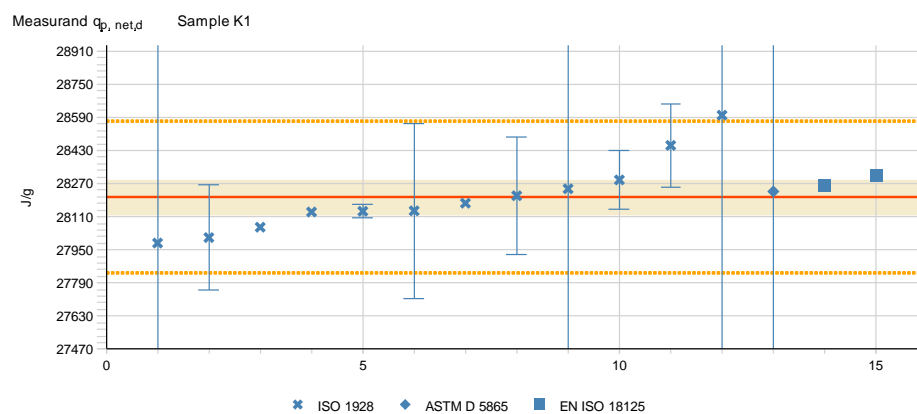
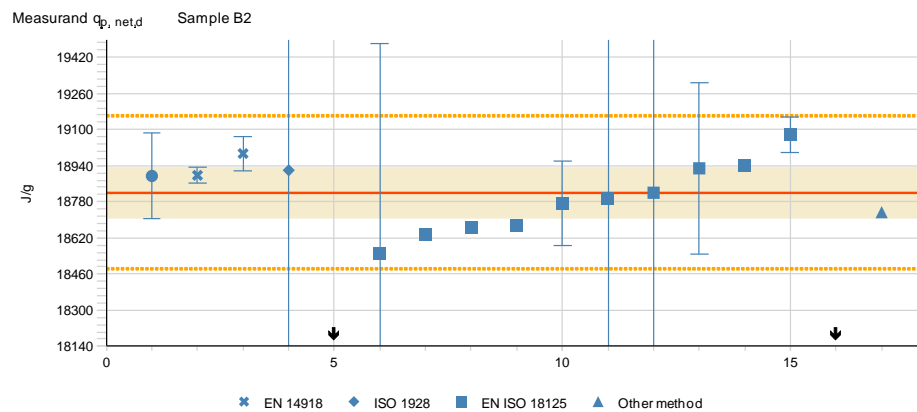
Measurand EF Sample K1

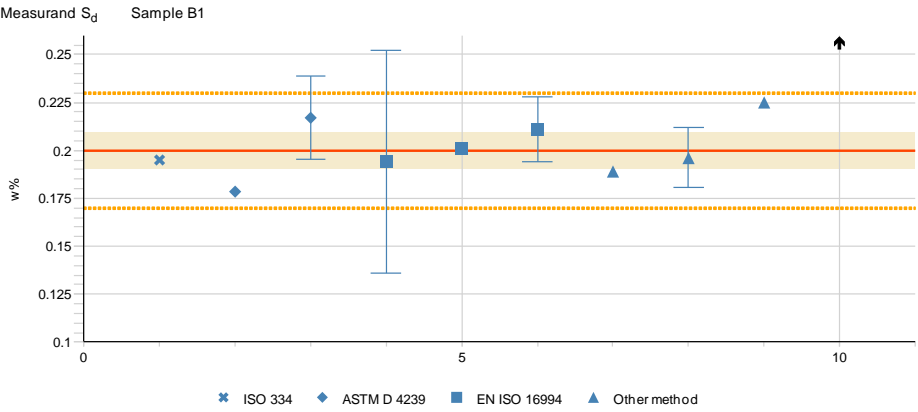
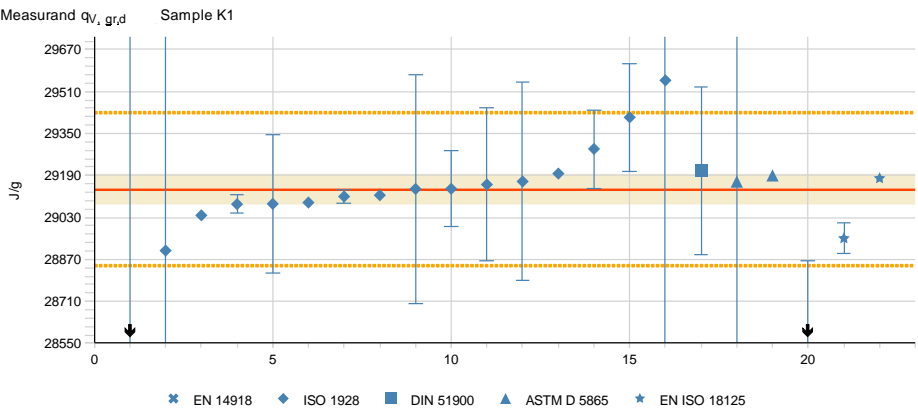
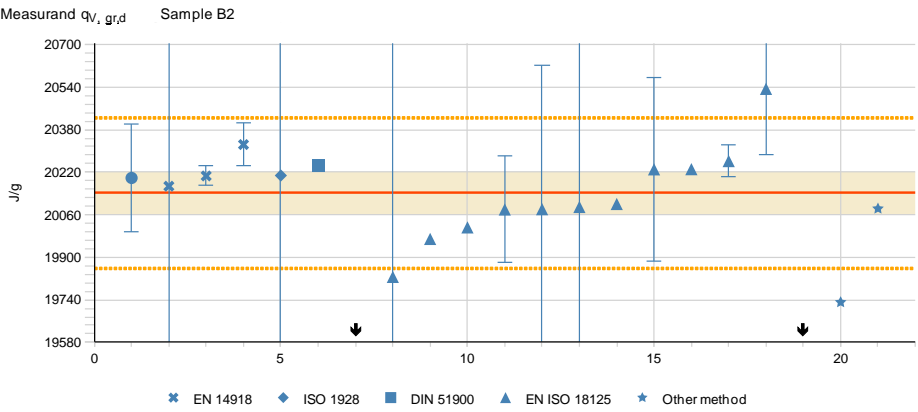
Measurand H_d Sample B1

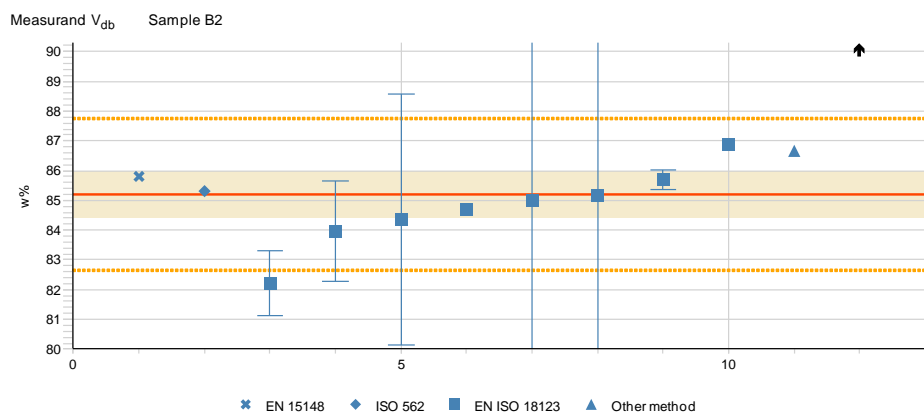
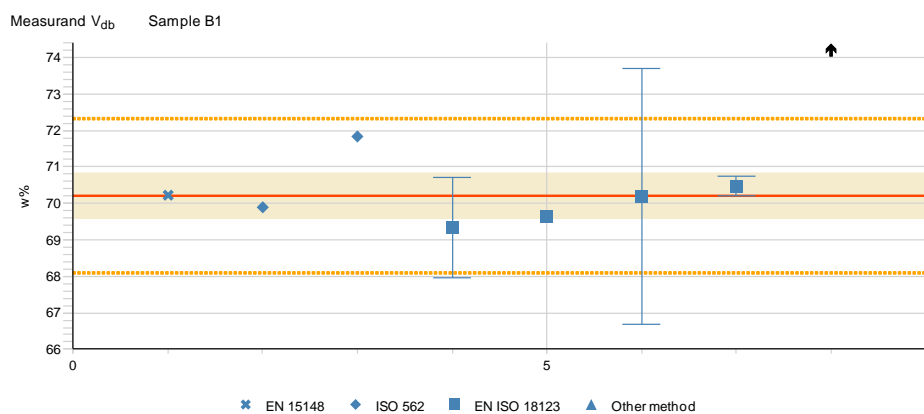
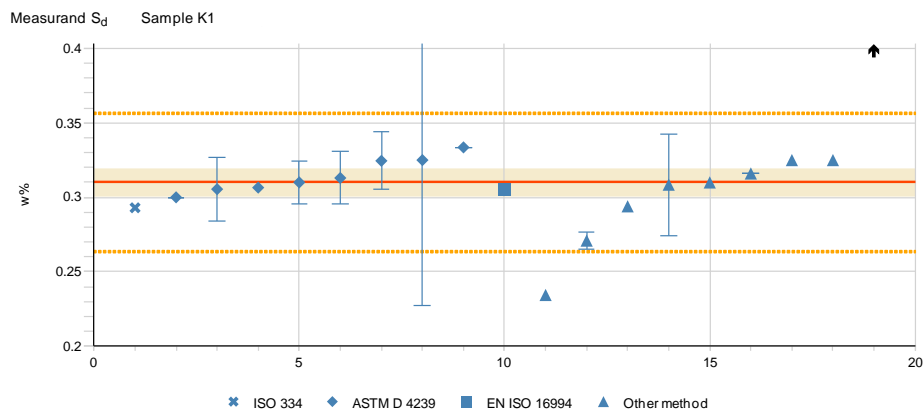


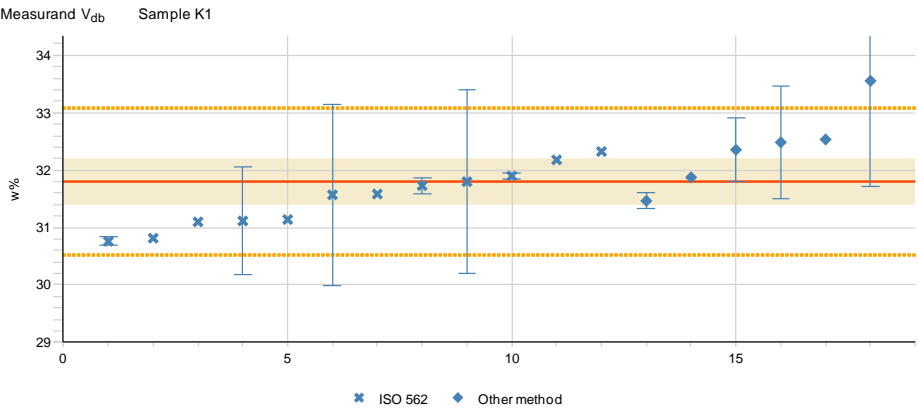
Measurand $M_{ad,d}$ Sample B2Measurand $M_{ad,d}$ Sample K1Measurand N_d Sample B1





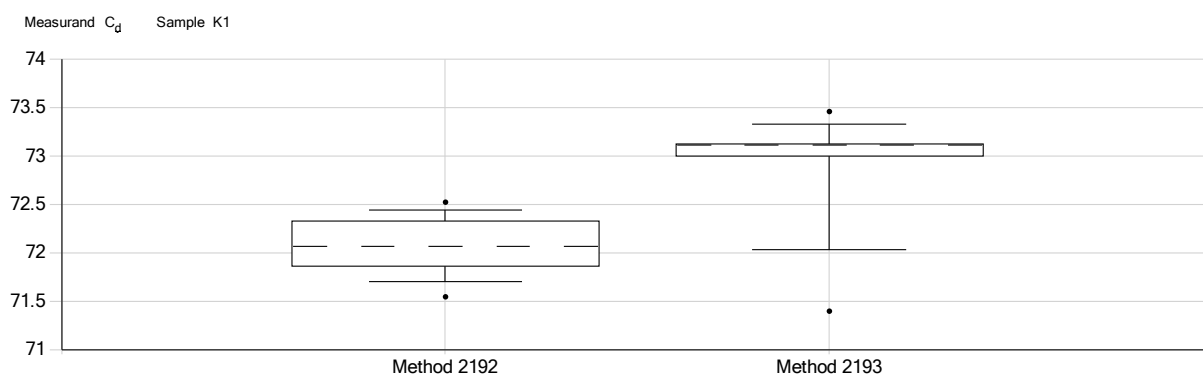






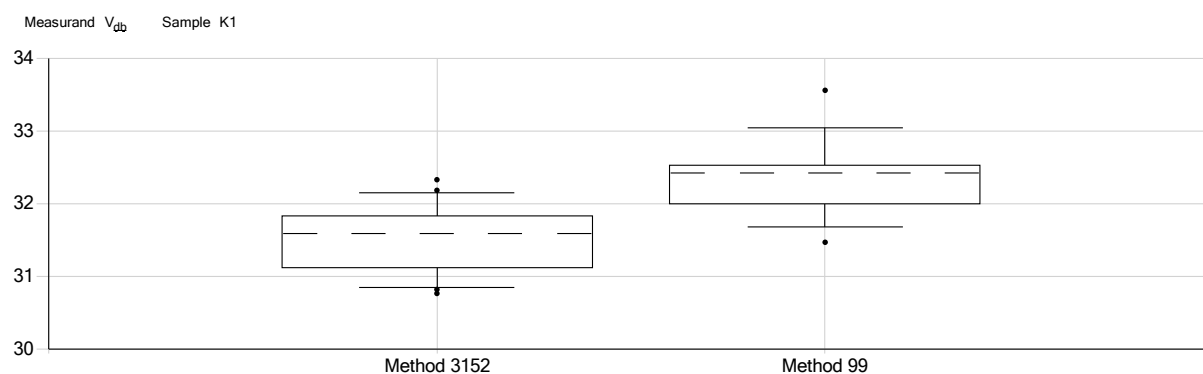
APPENDIX 13: Significant differences in the results reported using different methods

Boxplot figures: In the box the upper and lower limit included 50 % of the results. The dashed vertical line in the middle of the box is the median of the results. The vertical lines above and under the box describe the limits of 80 % of the results. The black dots describe the highest and smallest results within the center 90 % of the results.



C_d:K1 Method	n	Mean	Median	s
Method 2192: ISO 29541	8	72.1	72.1	0.3
Method 2193: ASTM D 5373	5	72.8	73.1	0.8

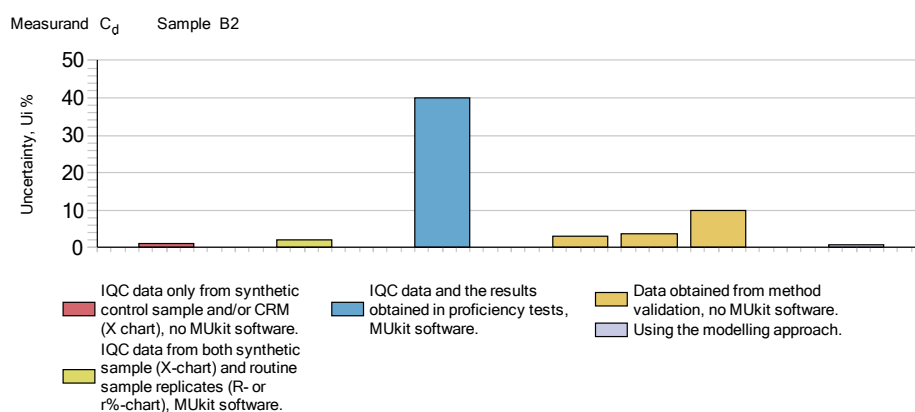
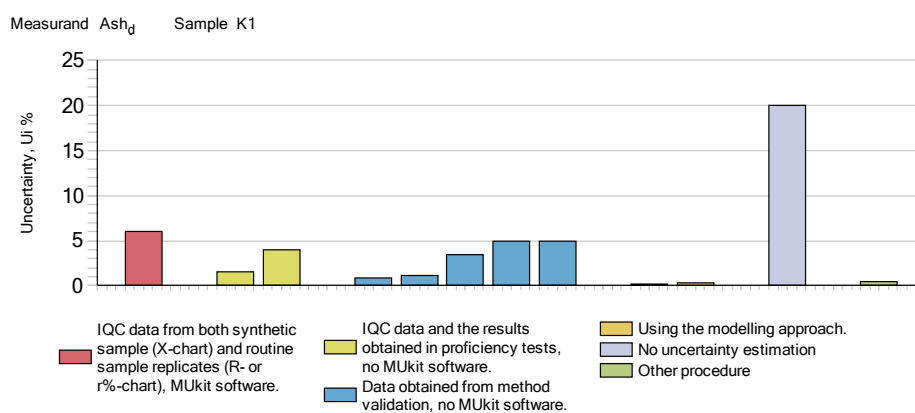
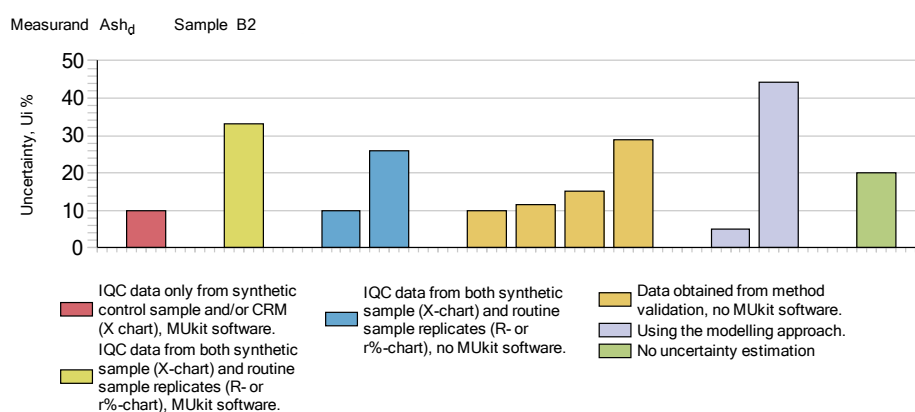
n: number of results. s: standard deviation

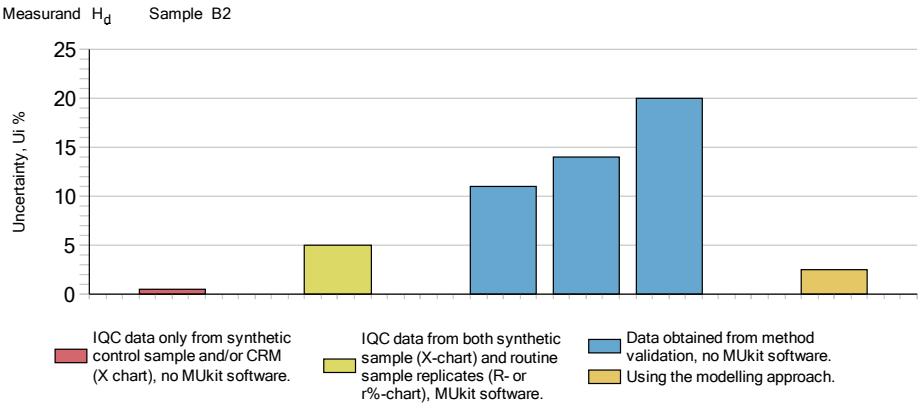
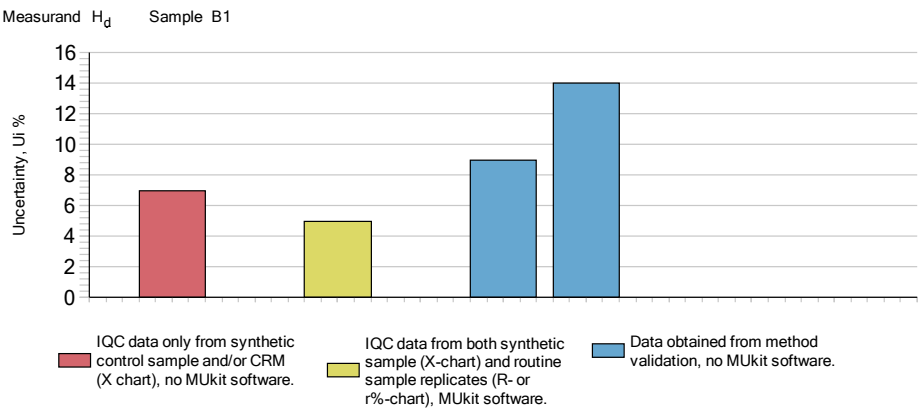
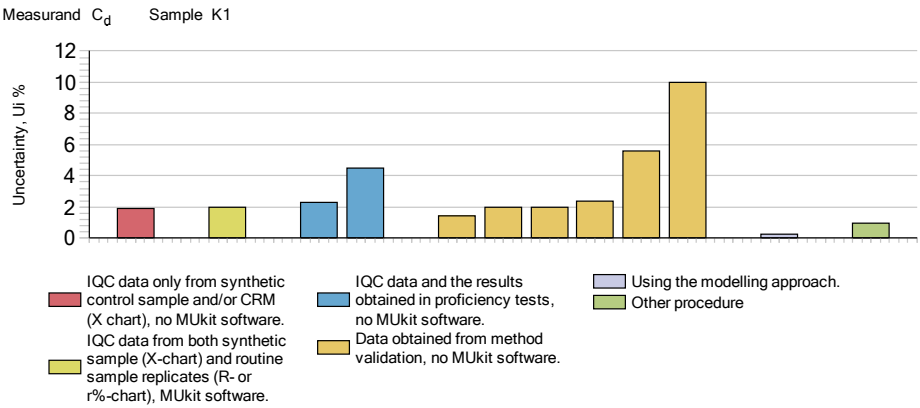


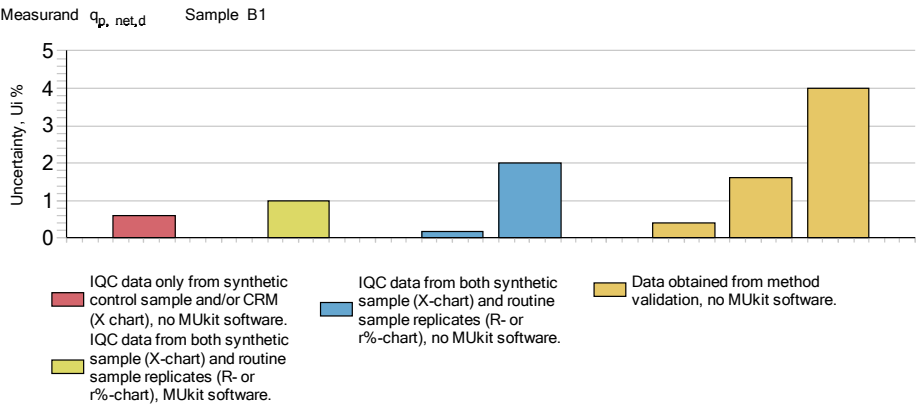
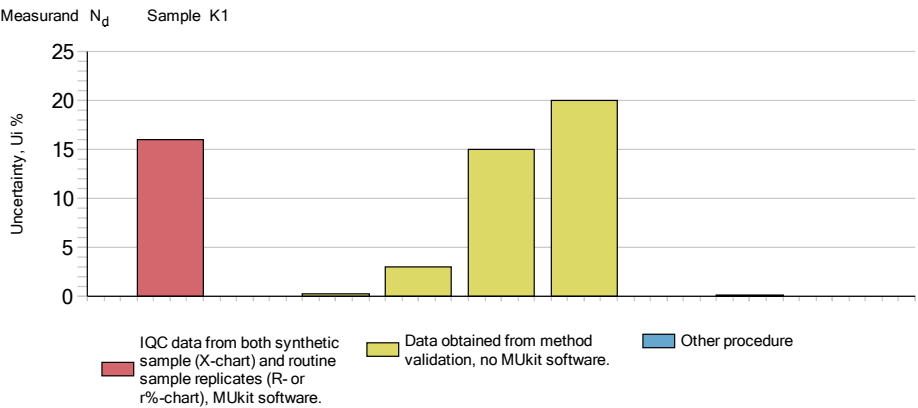
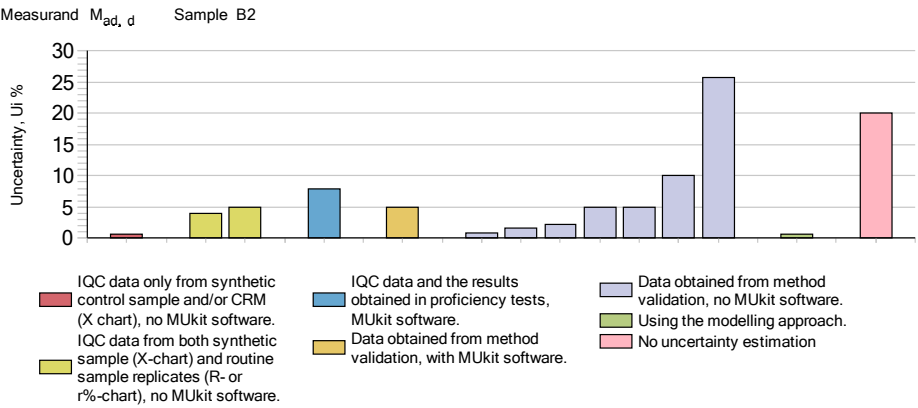
V_{db}: K1 Method	n	Mean	Median	s
Method 3152: ISO 562	12	31.5	31.6	0.5
Method 99: Other methods	6	32.4	32.4	0.7

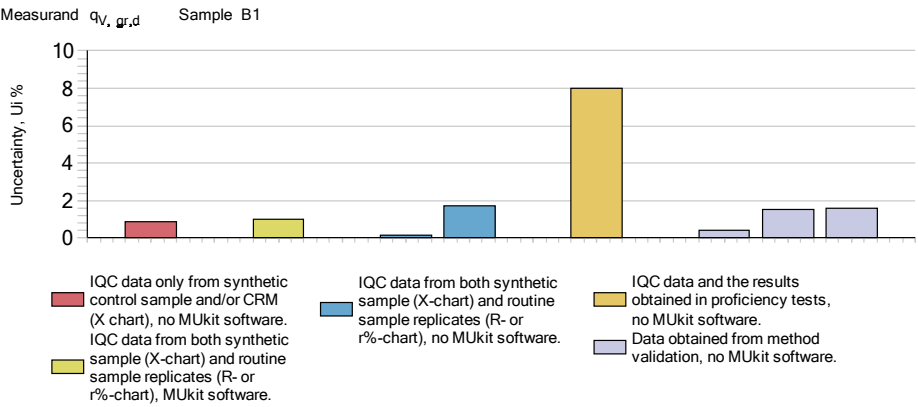
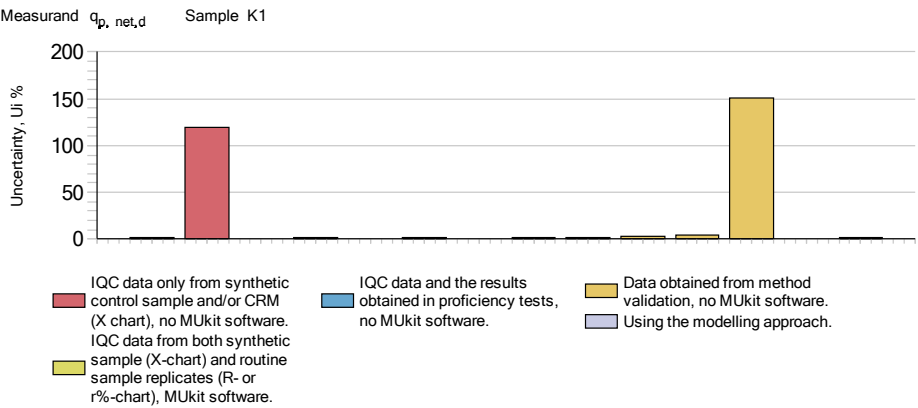
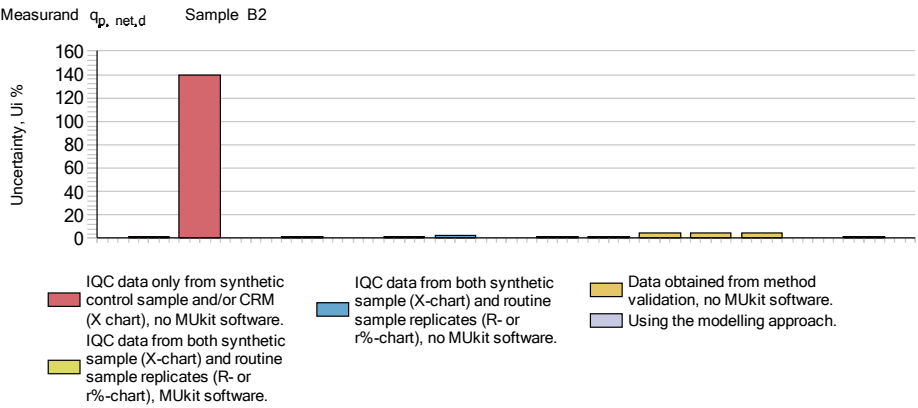
APPENDIX 14: Examples of measurement uncertainties reported by the participants

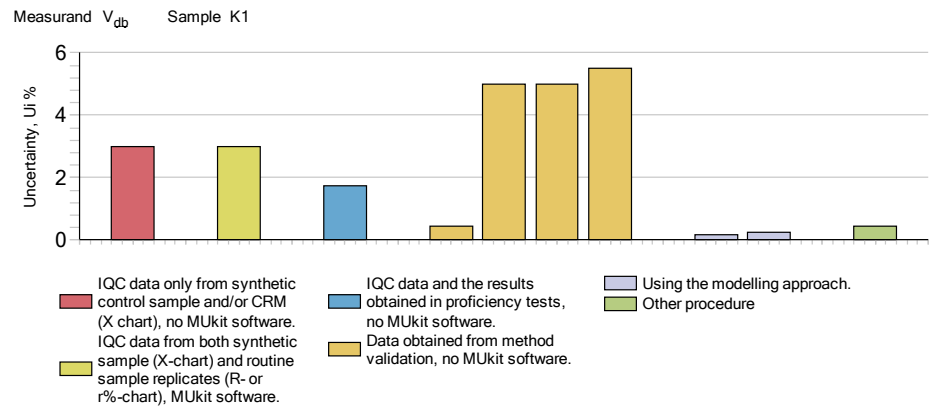
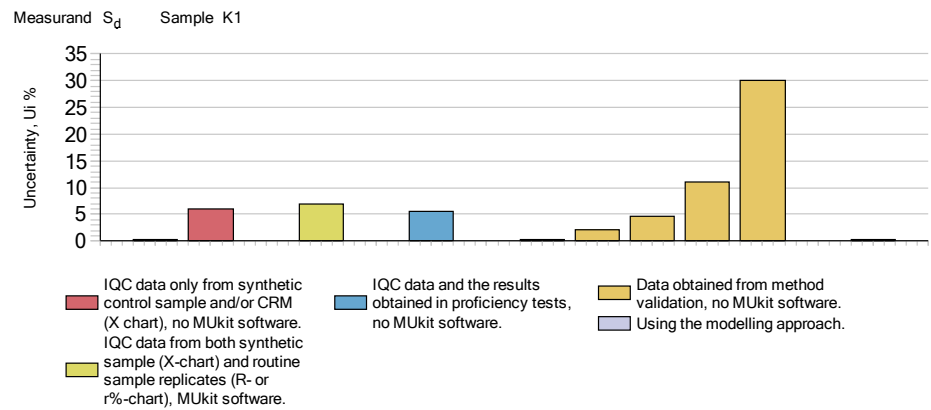
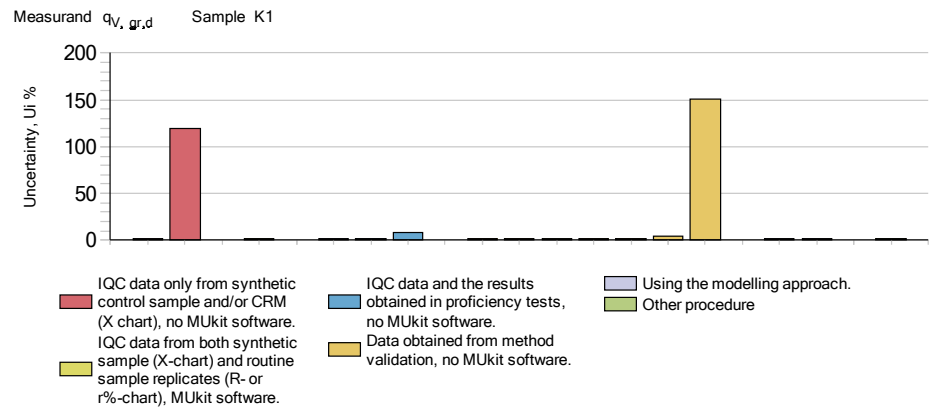
In figures, the presented expanded measurement uncertainties are grouped according to the method of estimation at 95 % confidence level ($k=2$). The expanded uncertainties were estimated mainly by using the internal quality control (IQC) data. The used procedures in figures below are distinguished e.g. between using or not using the MUKIT software for uncertainty estimation [31, 32] or using a modelling approach based [33, 34].













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